STABILITY AND MATURITY EVALUATION OF BIOSOLIDS COMPOST

By

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Compost stability and maturity are not well defined and there is no universally accepted method for their evaluation. The primary objectives of this study were to understand compost stabilization and maturation processes and develop a simple method for determining compost stability and maturity by studying extractable organic carbon. Storage effects on compost stability and maturity and effects of compost stability and application rate on C and N mineralization were also studied. Compost samples at different curing time were collected from biosolids compost facilities in Florida and Puerto Rica. CO_2 evolution and seed germination rate were tested for evaluation of compost stability and maturity. CO_2 evolution ranged 3.7-137 g kg^{-1} and seed germination rate ranged 7-95%. They were negatively correlated except for samples from one facility. WSOC was correlated ($p<0.01$) with both CO_2 evolution and seed germination rate.

CO_2 evolution rate of the most active compost was significantly reduced by storage especially by freezing. WSOC were reduced with air-dry and increased with freezing by up to 35% and 26% respectively. No storage method was recommended

among the tested methods since neither method was satisfactory in retaining the properties of fresh samples.

Compost carbon was sequentially extracted by water for 2h and NaOH for 2h and 24h and separated into humic (HA) and fulvic acid (FA). Each extracted OC was significantly correlated ($p<0.05$) to each other and linearly correlated to CO_2 evolution except for the water-2h extractable HA. Multiple regression showed that NaOH-24 h extractable OC was insignificant in contributing to CO_2 evolution. Water-2h and/or NaOH-2h extractable FA highly correlated (R^2=0.79 and 0.92) with CO_2 evolution and can be estimated by a simple photometric method for a large range of carbon concentration up to 4,000 mg L^{-1}.

Two compost samples from one facility were incubated with soil at three application rates over a period of 85 days. More CO_2 were released from the stable compost through out the incubation and from the low application rate treatment at early stage of incubation, indicating compost degraded faster with the lower application rate. Nitrification occurred earlier with stable compost and low application rate.

CHAPTER 1
INTRODUCTION

Statement of Problem

Interest in composting biosolids has greatly increased in the past decade (Goldstein, 1991; Goldstein and Steutevill, 1996). However, composted biosolids constitute only a small fraction of the biosolids produced annually (Goldstein, 1991, Goldstein and Block, 1999). This is also true for other solid wastes composting such as trimmed yard waste and municipal solid wastes. Compost quality is the key for the wide acceptance of compost by the public (Breslin, 1995; He et al., 1992; Inbar et al., 1990; Iannotti, 1994). Compost quality is usually characterized by its physical, chemical and biological properties, such as organic matter content, nutrient concentration, size distribution, water holding capacity, and organic and inorganic contaminants. In addition, compost stability and maturity are also an important indexes for compost quality, and these are of particular concerns when compost is used on agricultural soils (Mathur et al., 1993a).

Unstable or immature compost may cause adverse effects on plant growth through nitrogen immobilization, oxygen deficiency caused by strong microbial activity, and phytotoxicity of organic acids and ammonium (Breslin, 1995; Brodie, 1994; He et al., 1992; Inbar et al., 1990). Compost stability and maturity also have a great effect on carbon and nitrogen mineralization, an important process that must be understood for cost-effective and environmentally-safe use of biosolids compost (Hadas and Portnoy,

1994; Keeling et al., 1994; Bernal et al., 1998 a, c). As a result, much work has been done to understand stabilization and maturation processes of composting and to develop methods to evaluate the stability and maturity of end products (Hue and Liu, 1995; Iannotti et al., 1994; Inbar et al., 1991; Jimenez and Garcia, 1992; Mathur et al., 1993b). The definition and terminology concerning compost stability and maturity are still issues of scientific inquiry and debate today. There is no universally accepted official definition or methods for measuring compost stability and maturity. For this research, compost stability is defined by the microbial activity of compost and measured by the CO_2 evolution. Compost maturity is only related to phytotoxicity caused by insufficient composting. Phytotoxicity caused by other factors such as high concentration of salts is excluded.

Objectives

The overall objectives of this study were to: 1) compare several selected methods of evaluating compost stability and maturity, understand the difference and relation between the two concepts, and attempt to identify a simple and reliable method for determining stability and maturity of biosolids compost; 2) study impacts of storage methods on biosolids compost stability and maturity evaluation; 3) understand mechanisms of compost stabilization by exploring the relation between compost stability and extractable organic carbon; and 4) study the effects of compost stability and application rate on the C and N mineralization after application of compost to soil.

Compost Samples and Sampling Procedure

Compost samples used for this study were collected from four biosolids composting facilities in the State of Florida and one in Puerto Rica. The samples were at

the different curing stage of composting. The curing stage was operationally defined by each facility. The curing time for each sample, the composting method and other composting amendments used in each facility are listed in Table 1-1. The RC2 samples were from the same composting facility as RC, but collected at different time of year. The composition and ratios of biosolids to other feedstock materials differed among facilities but were relatively consistent within each facility. For each sample standing for a curing time, a composite sample consisting of three subsamples from three places which is approximately evenly distributed along the pile or bed of the same curing time was collected. At each place, a hole or a cut near the center of the pile was made and samples near the top, middle and bottom of the pile were collected and combined. Compost within about half foot on the surface and very bottom were avoided. The samples taken from the different places at each pile or bed were then combined. Curing piles from each facility had different heights, thus the depths at which samples were collected were different too.

Table 1-1. Location of composting facility, composting methods and amendments used in each facility and curing time of collected biosolids samples.

Sample	Curing Time	Location	Composting method	Other wastes and amendment	Ratio of biosolid to other waste
RC-1 RC-2 RC-3	0 7 30	Reedy Creek, Orlando	Force aerated windrow	Food waste, yard waste, animal manure, & wood chips	Biosolids and other wastes to wood chips at 1:3.5 by volume
RC2-1 RC2-2 RC2-3 RC2-4	0 9 21 25	Reedy Creek, Orlando	Force aerated windrow	Food waste, yard waste, animal manure, & wood chips	Biosolids and other wastes to wood chips at 1:3.5 by volume
WPB-1 WPB-2 WPB-3	0 7 30	West Palm Beach	In vessel (IPS)†	Ground yard waste	Biosolids to yard waste 1:1 at 40% dry solids
SA-1 SA-2 SA-3 SA-4 SA-5	0 7 14 26 35	Sarasota	In vessel (PURAC)‡	Sawdust	Biosolids to sawdust 1:2.5 by dry mass
MW-1 MW-2	30 90	Meadow Wood	Windrow	Yard waste	Biosolids to yard waste at ratio 1:3 by volume
PR-1 PR-2 PR-3	0 7 14	Puerto Rica	Windrow	Yard waste	N/A

† Agitated bed technology by International Process Systems (IPS).
‡ Closed in vessel composting system developed by PURAC Engineering, Inc.

CHAPTER 2
LITERATURE REVIEW

Methodology of Evaluating Compost Stability and Maturity

Compost stability and maturity are used frequently in the literature, sometimes interchangeably and without clear definition. They both indicate transformation of nutrient containing materials to a stable organic matter, and stable/mature compost will not incur adverse agricultural and environmental impacts upon land application (Brinton, 1996; Brodie et al., 1994; Jimenez and Garcia, 1989). Some researchers consider stability and maturity as two different indexes (Frost et al., 1992; Iannotti et al., 1993). They define compost stability as the degree to which organic materials have been decomposed, and is a function of microbiological activity (Iannotti et al., 1993; Chen and Inbar, 1993). Compost maturity, on the other hand, is a more elusive concept and generally refers to the development of beneficial effects on plants after compost utilization (Frost et al., 1992). Such beneficial effects, resulting from complete decomposition of phytotoxic organic substances, is determined with plant response (Iannotti et al., 1993). On the other hand, compost stability and maturity are closely related and both depend on decomposing stages and forms of different chemical constituents present in compost. Consequently, biological, chemical and physical methods are all valuable in evaluating compost stability and maturity.

Evaluation of Compost Stability

Respirometric method

Respiration rate, based on CO_2 evolution or O_2 consumption, reflects the microbial activity of compost. Combined with a plant test, respiration rate can empirically define the respiratory rate corresponding to compost stability (Morel et al., 1985). The traditional respirometer, commonly called a Warburg instrument, measures CO_2 evolution by absorption by NaOH solution and the corresponding change in pressure is measured by a manometer at a constant temperature and gas volume (Haug and Ellsworth, 1991). This method is limited to relatively small, homogeneous samples and requires a relatively long incubation time. The O_2 consumption method is receiving more interest recently to quantify compost stability (Iannotti et al., 1994). An easy and reliable way to measure O_2 consumption is to use an O_2 sensitive probe (Frost et al., 1992; Iannotti, et al., 1993; 1994). Lasaridi and Stentiford (1998) described an automated procedure employing a dissolved oxygen probe to assess compost stability.

Source materials with little decomposition may have low microbial activities, producing misleading results (Seekins, 1996). Other factors affecting respirometric measurement include compost moisture content, temperature control, and potential toxic agents. Lasaridi and Stentiford (1998) found that measuring the respiration rate of an aqueous compost suspension rather than a solid compost sample has certain advantages, such as the test is not affected by variations in the water potential of the samples and there is immediate contact between substrate, microbes and oxygen leading to maximum reaction rates.

Self-heating test

Dewar method is a standardized procedure to evaluate self-heating. Using a vacuum lined vessel called Dewar vessel, this method is to precisely record the highest temperature achieved after placement of compost into the vessel for several days. This method is easy to use and inexpensive. However, compost density and moisture content greatly affect the results due to the high specific heat of water (Iannotti, et al., 1993). As a result, it is usually used only as a complementary measure (Seekins, 1996), since the method alone is insufficient to predict compost stability.

Microbial population evolution

Microbial populations change continually during aerobic composting (Poincelot, 1977). Even though the overall level of microorganisms may not change markedly, shift in subpopulations sequentially degrade the various substrates (Atkinson et al., 1996). Fungi are believed to be the primary microorganisms capable of degrading the recalcitrant substrate such as cellulose, ligno-cellulose and lignin (Atkinson et al., 1996; Herrmann and Shane, 1996). If the dominant population shifts to fungi, it indicates the compost is approaching stability. But it is difficult to relate the compost stability to the biomass or activity level of fungi because of hyphal fragmentation and spore formation (Atkinson et al., 1996). The ammonia-producing bacteria and autotrophic nitrifying bacteria are suggested as indicators of compost stability. Riffaldi et al. (1986) found ammonia-producing bacteria increased during the first phase of composting, reaching their maximum within 15 days, then suddenly reduced to below the detection limit by the 60th day of composting. In contrast, nitrifying bacteria reached the maximum activity by the 80th day of composting and were still present at the last sampling day (140^{th} day).

Other biochemical parameters

Enzymatic activities indirectly reflect the microbial community and the substrates being utilized, therefore possibly indicating compost stability. Some extracellular hydrolases, mainly urease, phosphatase, protease and β-glycosidase, usually exhibit high activities at the beginning of composting and low activities in stabilized organic matter (Ayuso et al., 1996; Garcia et al., 1993). Dehydrogenase activity has been found highly correlated to temperature in composting pile which indicates the microbial activity (Tiquia et al., 1996). However these enzymes may behave quite differently in composts from different sources (Diaz-Burgos et al., 1993). Cellulase activity increases when the majority of simple organic compounds are deplete (Herrmann and Shann, 1993; Riffaldi et al., 1986); consequently, it may be a good indicator of compost stability. On the other hand, Herrmann and Shann (1993) found lipase activity measured on a 10-carbon-length lipid substrate was low throughout the active composting process and most of the curing process, but extraordinarily high in the mature compost. The ATP level in compost reflects microbial biomass. It decreased markedly during the earlier stages of composting, then stabilized to a low level (Garcia et al., 1992c). It was highly correlated with folin-reactive compounds and hydrolysable polysaccharides, which probably comprise the major available C and N sources for microbial growth (Diaz-Burgos et al., 1993). However, ATP measurement requires sophisticated technical skills and instruments (Mathur et al., 1993a).

C/N ratio in solid phase

This is the most frequently and widely used criterion to estimate the degree of compost stability and define compost agronomic quality. The ideal C/N ratio for a mature compost is 10-20, reflecting the C/N ratios of different forms of soil humus

(Jimenez and Garcia, 1992; Mathur et al., 1993a). A stable compost requires no soil N for further decomposition of the product, but releases mineral N into the soil. C/N ratios of up to 20 are therefore acceptable for composts as long as they are stable, especially for compost with high lignin content or compost of woody recalcitrant materials. However, C/N ratios below 20 can often be found in raw materials, such as sewage sludge or chicken manure (Chanyasak et al., 1983a; Hue and Liu, 1995). On the other hand, among four cattle manures with different C/N ratios (9-18), the one with low C/N ratio not the manure having high C/N ratio was found to cause N immobilization (Nyamangara et al., 1999). As a result, when C/N ratio is used to indicate compost stability, type of source materials and initial C/N must be considered (Jimenez and Garcia, 1989; Mathur et al., 1993a; Morel et al., 1985). C/N ratio alone thus is insufficient to indicate compost stability.

C/N ratio in water extract

C/N ratios in water extracts change more significantly than C/N ratios of solid samples. Chanyasak et al. (1982) noted that in mature municipal solid waste compost, C/N ratio of water extract declined to 5-6, which was confirmed by other investigators (Garcia et al., 1992a; Hue and Liu 1995; Jimenez and Garcia, 1992). These authors proposed to use this parameter as an essential indicator of compost stability. The problem with this parameter is that the organic N in water extract of certain compost may be too low to be determined precisely (Hue and Liu, 1995).

Cation exchange capacity (CEC)

As humification progresses during composting, the CEC increases and significantly correlates to the C/N ratio of solid material (Jimenez and Garcia, 1992). Total dry matter may not be suitable as the calculation basis for various composts

because the CEC mostly resides in the organic fraction. Even measured as cmol/kg of organic matter, Jacas et al. (1987) found CEC ranged from 32.1 to 63.3 among 8 stable composts. A much greater variation was found by Estrada et al. (1987) among 25 composts, ranging from 46.4 to 153. The variations arise from the fact that not all of the organic matter is humus (Mathur et al., 1993a). The CEC values for compost stability cannot be extrapolated from one kind of material to another (Bernal et al., 1996). In addition, CEC measurement is a rather time consuming procedure.

NH_4^+ and NO_3^-

Complex N-compounds are degraded to amino acids and ammonia during the early stages of composting. As a result, ammonia concentration is as high as a few hundred mg kg^{-1} at the early stages of composting, and then decreases sharply to a stable value of about 50 mg kg^{-1} in a municipal solid waste (MSW) compost (Avnimelech et al., 1996). On the contrary, the organisms that oxidize NH_4^+ to NO_3^- are only active in moderate temperatures. As a result, low level of NH_4^+ and high level of NO_3^- in a compost could be a sign of stability, but no particular level of NO_3^- or its ratio to NH_4^+ can be relied upon as an indicator of compost stability (Diaz and Trezek, 1979; Grebus et al., 1994).

Water extract analyses

Chanyasak et al. (1982) and Hirai et al. (1986) measured amino acids, volatile aliphatic acids (low molecular fatty acids), peptides and polysaccharides in water extracts of raw materials and their composts. The water extracts of raw materials and immature composts always contained more aliphatic acids and amino acids than the extracts of mature composts on an equivalent total dry weight basis. The water extracts of mature

compost from sewage sludge contained more polysaccharides and peptides than extracts of the raw material prior to composting. They also found that water extracts of mature composts always contained less total organic carbon than did extracts of the corresponding raw materials or of immature compost. Hue and Liu (1995) confirmed this trend, and proposed that a compost is stable when water-soluble C is less than 10 g/dg of dry matter. Mathur et al. (1993b) recommended the absorption of water extract at 665 nm as a simple and reliable method to evaluate compost stability.

Humic substance evolution

Traditionally, humus formed by composting is extracted with diluted sodium hydroxide, or sodium pyrophosphate or a combination of these two. The fraction that precipitates at pH=1-2 is referred to as humic acid (HA), whereas the soluble fraction at pH=2 contains fulvic acid (FA). Humus (FA+HA) content of a compost expressed as percent of total organic matter is called extraction rate. This value would be expected to increase with composting. However Garcia et al. (1991a) found the extraction rate decreased during the composting process, because the extractable carbon includes non-humus fraction, particularly in the initial stages of composting. The fact that extraction rate is not indicative of organic matter stability has been confirmed by several investigations (Bernal et al., 1996; Hanninen et al., 1995; Jimenez and Garcia, 1992). The ratio of humic acid (HA) to total organic matter expressed as humification index (HI) decreased as compost stabilized (Bernal et al., 1996), but the differences between raw material and mature compost were not sharp for some composts (Garcia et al., 1991a; Saviozzi et al., 1988). Another parameter proposed for assessing organic matter stability is the carbon content ratio of HA to FA, referred to as the polymerization ratio. This ratio

also varies among various composts (Bernal et al., 1996), and sometimes fails to exclude the raw material from compost (Hue and Liu, 1995).

Spectrometric analysis of organic matter

Hoping that more sophisticated methods may reveal maturation of compost humic substances, Inbar et al. (1990), Chen and Inbar (1993) and Chefetz et al. (1996) studied compost samples by carbon-13 nuclear magnetic resonance (^{13}C-NMR) and Fourier transform infrared (FTIR). The results revealed only slight compositional and structural changes between raw material and mature composts.

Non-humic organic fractionation

The content of lignin has long been used to estimate the biodegradability of organic waste. Peeninck and Verdonck (1986) proposed to use it as an index for compost stability. But it is only suitable for wastes of high lignin or cellulose-lignin compounds, not for sewage sludge, or food waste. Dinel et al. (1996) developed an organic matter stability test by sequential extraction of lipid with diethyl ether (DEE) and chloroform ($CHCl_3$) and comparison of their concentration with total lipid. Hanninen et al. (1995) emphasized the role of carbohydrates because they are covalently bound to the structures of FA and HA. It would be impractical to use polysaccharide content as a measure of compost stability, because not all polysaccharides decompose at equal speed and polysaccharides are also synthesized by some microorganisms during the compost maturation phase, especially for sewage sludge compost. Determination of a readily decomposable polysaccharide, such as starch, is only suitable for homogenized wastes with a significant amount of starches (Mooijman and Lustenhouwer, 1987).

Evaluation of Compost Maturity (Phytotoxicity)

Plant bioassay

Plant bioassay is perhaps the most appropriate test for maturity because it directly indicates when a compost may be used without inhibitory effects. Usually, plant growth in mature compost is similar or higher than the control. The early stage of plant growth is more sensitive to immature compost (Henry and Harrison, 1996). The inhibitory effects of immature compost can be also caused by low molecular organic compounds such as volatile fatty acids or high concentration of ammonia (Chanyasak et al., 1983 a, b; Edney and Rizvi, 1996; Shiralipour et al., 1997; Wong, 1985). These unfavorable effects of immature compost can be eliminated by further curing. Other factors, such as a high concentration of soluble salts, high level of heavy metals or pesticide or other toxic substances, will also affect plant growth but cannot be eliminated by further curing (Iannotti et al., 1994). These factors may affect the results of plant test for maturity. In addition, a plant bioassay is time a time consuming procedure, usually taking 2 or 3 weeks.

Seed germination test

A germination test may be considered as a simplified version of a plant bioassay. It takes less time and demands less labor. Zucconi et al. (1981) incubated a water extracts of compost with cress, and the percent of seed germinated and length of roots were determined as germination indices. Based on this idea, Mathur et al. (1986) developed a simpler method by using solid compost directly. The germination method can detect compost toxicity without considering any beneficial phytoalexin or nutritional effect of the compost on root growth (Mathur et al., 1993a). But it is far different from

the field condition. The results can also be misleading if herbicide residues, high salinity or other potential phytotoxic agents are present (Iannotti et al., 1994).

Volatile fatty acid analysis

Several studies had shown considerable concentrations of fatty acids form during composting of organic wastes, especially at early stage of decomposition (Haner et al., 1994; Kirchmann and Widen, 1994). Slow or complete stop of plant growth in immature compost with high concentration of fatty acid indicates the presence of phytotoxins (Wong, 1985). Chemical analysis of fatty acid may help to reveal the toxic levels of immature compost (Devleeschauwer et al., 1981), but phytotoxicity may not depend on any single fatty acid. Instead it may depend on a combination of various organic acids, which may render results of chemical analyses difficult to interpret.

Carbon and Nitrogen Mineralization in Soils Amended with Compost

Beneficial use of organic wastes depends on identifying a management strategy that supports crop production and protects the environment. Nitrogen is an element of major research interest because organic wastes, especially untreated wastes, can provide substantial amount of N to plant growth, but at the same time it may pose a great potential of nitrate contamination for ground and surface waters. From the point of view of N management, composting serves two major purposes: 1) to reduce C/N in order to minimize potential N immobilization, and 2) to stabilize the N so that this nutrient can be slowly released to reduce the potential risk of nitrate leaching. However, immobilization of N has been frequently reported with application of unstablized compost, especially at the early stages of an experiment despite some low C/N ratio. Leaching of excess nitrate may also occur with compost application, especially when applied at high rate or at a

time of slow crop growth, or when mature compost contains a high concentration of nitrate. In a compost amended soil, the balance between N immobilization and mineralization is very delicate and influenced by many factors. N dynamics are usually studied together with C because these two elements are so closely interrelated.

Compost sources, composting process, and physical and chemical characteristics may all greatly affect the degradability of C containing organics and N availability. In a column study, He et al. (2000) found the organic N mineralization rates for biosolids, yard trimming and a biosolid/yard trimming co-compost were 23.3, 23.5 and 48.4%, respectively, during a 1-year incubation. The recovered mineral N accounted for 36, 43 and 57% of the total mineralized N. During the first 6 months NH_4-N was the dominant form of mineralized N, but NO_3-N accounted for more than 50% of the total mineral N during the later part of the incubation for yard waste and biosolid. NO_3-N was the dominant mineral N throughout the whole incubation period for the compost. Kirchmann et al. (1991) compared C and N mineralization of fresh, aerobic and anaerobic treated manures from cattle, pig and poultry in a 2-month incubation. The rates of CO_2 evolution from fresh manures was higher than from aerobically treated manures but lower than from anaerobically treated manures, independent of the animal species. With the exception of fresh poultry manure, which has a high content of easily decomposable uric acid N, fresh and aerobic manures showed a very small net mineralization of N or even immobilized N. In anaerobically treated manures, between 50-70% of the total N was present as NH_4-N, which resulted in a high inorganic N content in soil compared to fresh and aerobic manures.

C/N ratio is a very important parameter that affects N dynamics in the soil. In a 1-2 month incubation of soil with four organic amendments and inorganic N of different stability, Jedidi et al. (1995) found that N mineralization was correlated with C/N ratios (R^2=0.936). However, in an effort to control N immobilization by adding inorganic N, Nyamangara et al. (1999) found N immobilization occurs in manures with the lowest C/N ratio, instead of the high C/N ratio. Their conclusion was that it is not possible to use the C/N ratio of aerobically decomposed cattle manure as a tool to predict mineralization or immobilization of N.

CHAPTER 3
COMPARISON OF METHODS FOR EVALUATING STABILITY AND MATURITY OF BIOSOLID COMPOST

Introduction

As stated earlier, the terms "compost stability" and "compost maturity" are frequently used in the scientific literature. While stability and maturity were sometimes treated as different terms for the same compost property with no real distinction, some researchers have tried to differentiate compost stability and maturity because differences between the two have been noted. Generally, the term "compost stability" is better understood, and a well accepted definition is "the rate or degree of organic matter decomposition". As such, compost stability can be expressed as a function of microbiological activity; it can be determined by O_2 uptake rate, CO_2 production rate, or by the heat released as a result of microbial activity (Iannotti et al., 1993; Chen and Inbar, 1993). "Compost maturity" generally refers to the degree of decomposition of phytotoxic organic substances produced during the active composting stage and usually is assessed by plant response or seed testing (Zucconi et al., 1981; Iannotti et al., 1993). Understanding and properly defining compost stability and maturity will assist standardization and regulation of the methods used to evaluate compost quality.

Stable or mature composts have been reported to contain less water soluble organic carbon (WSOC) than the corresponding raw materials or immature composts (Chanyasak et al., 1980; Hirai et al., 1986; Hue and Liu, 1995; Mathur et al., 1993b). Furthermore, Chanyasak et al. (1980) reported that the WSOC of immature compost

could contain both humic substances and non-humic substances. Chefetz et al. (1998b) found that the percentage of WSOC made of humic-like substances increased during the late stages of composting. Since high absorbance is one of the most significant characteristics of humic substances (Orlov, 1995), the change of absorbance of water-extractable organic matter may reflect the degree of humification and stability of compost. It is, therefore, possible to assess compost maturity/stability by determining both WSOC concentration and its spectrophotometric properties. In addition, compost stability maturity depends on the chemical constituents present in a compost feed stock as well as those present in various decomposition stages. Thus, compost chemical properties and feedstock are both potentially important in evaluating compost stability and maturity.

By comparing several chemical and biological parameters related to compost stability and maturity properties, the objectives of this research were to 1) examine relatively simple parameters to monitor the stabilization and maturation processes of biosolid compost with different feedstock materials; 2) compare and determine if compost stability based on respiration rate and maturity based on seed germination rate are different properties and if both measurements are needed for compost quality control; 3) determine if WSOC concentration and its spectrophotometric properties are suitable for evaluating compost stability and maturity.

Materials and Methods

Sample Handling and Chemical and Physical Analysis

For this part of study, samples from RC, WPB and SA were used. Collected samples were placed in polyethylene bags, packed on ice in a cooler and shipped to the

lab the same day. Upon arrival, the samples were sieved through a 9.5 mm screen to remove large particles and kept refrigerated until analysis (The U.S. Composting Council, 1998). A sub-sample was air-dried and ground to pass through a 2 mm screen for chemical analyses. Fresh samples ere used to determine compost moisture content, water holding capacity, pH and electrical conductivity (EC). Moisture content was determined as weight loss upon drying at 105 °C in an oven for 24 h. Water holding capacity was estimated at 0.01 MPa (10^6 Pa) pressure (Cassel and Nielsen, 1982). Electrical conductivity and pH were determined from a 1:10 (Tiquia and Tam 1998; Rajbanshi et al., 1998) dry compost/ water extract ratio using an Accumet pH/Conductivity meter (Model 20). Total volatile solids (TVS) were determined as sample weight loss (previously oven-dried at 105 °C) upon ashing at 550°C for 4 h in a muffle furnace. Total organic carbon was calculated by dividing the TVS values by 1.76 (Nelson and Sommers, 1982). Compost samples were digested for total Kjeldahl nitrogen (TKN) and total P analyses (Bremner and Mulvaney, 1982). TKN was analyzed using an Alpkem air-segmented, continuous-flow, automated spectrophotometer. Total P was measured colormetrically using a Shimadzu UV-160U Spectrophotometer (Olsen and Sommers, 1982).

CO_2 Evolution

Microbial respiration of compost samples, based on CO_2 evolution, was measured using a modified procedure of Iannotti et al. (1994). Approximately 10 g of screened sample at 60% (w/w) moisture content was sealed in a 0.5 L vessel along with a beaker containing a known volume of 0.5 N NaOH solution. Samples were randomized and incubated for 7 to 8 d at room temperature (24±2°C). During the incubation, the released

CO_2 was captured by the NaOH solution, which was then analyzed titrimetrically at regular intervals.

Seed Germination Test

A modified phytotoxicity test employing seed germination was used (Zucconi et al., 1981). No. 2 Whatman filter paper was placed inside a 15x100 mm sterilized and disposable Petri dish. The filter paper was wetted with 9 mL of 1:10 compost to water extract and 30 tomato seeds (*Lycopersicon esculentum* L.) were then placed on the paper. Deionized water was used as a control and all experiments were run in triplicate. The Petri dishes were sealed with Parafilm to minimize water loss while allowing air penetration and were then kept in the dark for 4 d at room temperature. At the end of 4 d, the percentage of seed germination in compost extract was compared to that of the water control. A preliminary test on the effects of soluble salts on tomato seed germination using $CaCl_2$ and NaCl solutions revealed no inhibition of seed germination when solution EC was < 0.5 S m^{-1}.

Water Soluble Organic Carbon

Water soluble organic carbon was determined by first extracting a moist compost sample with deionized distilled water (DDW) (water to solid ratio of 10:1) for 2 h in a horizontal shaker at room temperature. The suspension was then centrifuged at 9,630 g for 10 min and filtered through a 0.45 μm membrane filter. The filtrates were used for WSOC analysis. An aliquot of the filtrate was adjusted to pH<2 using 6 N HCl and then centrifuged at 9,630g 10 min and filtered through a 0.45 μm membrane filter. The precipitate pellet collected at the bottom of centrifuge tube was water-extractable humic

acid (HA), while the filtrate contained fulvic acid and other nonhumic substances (FA) (Swift, 1996).

Organic carbon concentrations of total WSOC, FA and HA fractions were determined using a Shimadzu TOC-5050A (Columbia, MD) carbon analyzer. In addition, the absorbance of WSOC, HA and FA solutions at 420 nm were measured using a Shimadzu UV160U UV-visible spectrophotometer with samples diluted to ~ 500 mg C L^{-1} for FA and 100 mg C L^{-1} for HA solutions. The readings were then used to calculate the mass specific absorbance (MSA) which is defined as the absorbance per mass unit of WSOC (L $mg^{-1}m^{-1}$).

Statistical Analyses

The SAS (release 6.12) procedure GLM with LSMEAN and PDIFF options was used to compute the p value of statistical differences between samples (SAS, 1987). Since compost stability and maturity differed greatly from one facility to another, samples from each facility were treated as nested by facility. Correlation coefficients between parameters were calculated using a CORR procedure.

Results and Discussion

Physical and Chemical Properties of Compost

The physical and chemical properties of the compost samples varied greatly from one facility to another (Table 3-1). The moisture content (dry weight basis) and the water holding capacity of the RC facility samples were relatively low compared to the other two because the RC facility uses an aerated static-pile system for composting relative to the closed system used in the other two composting facilities. In order to recycle the wood-chip bulking agent and improve compost recovery from the screening process

Table 3-1. Select physical and chemical properties of tested biosolids compost samples.

Sample	Curing (d)	Moisture content ($g\ kg^{-1}$)	WHC† ($g\ kg^{-1}$)	pH (water)	Electrical conductivity ($S\ m^{-1}$)	Volatile solids ($g\ kg^{-1}$)	TKN ‡ ($g\ kg^{-1}$)	Organic Carbon ($g\ kg^{-1}$)	C/N ratio
RC-1	0	654	1148	5.8	0.48	681	28.4	398	14
RC-2	7	585	1116	7.0	0.45	737	25.8	413	16
RC-3	30	507	991	8.4	0.38	668	26.1	392	15
WPB-1	0	693	1279	8.4	0.18	562	16.8	319	19
WPB-2	7	601	1075	8.7	0.19	614	18.0	342	19
WPB-3	30	1101	1312	8.8	0.16	598	18.2	346	19
SA-1	0	1039	1468	7.3	0.13	914	13.6	517	38
SA-2	7	950	1454	7.6	0.13	911	13.4	522	39
SA-3	14	1078	1423	7.8	0.14	916	14.1	522	37
SA-4	26	1001	1342	8.0	0.16	908	12.8	512	40
SA-5	35	914	1428	7.0	0.14	902	13.8	511	37

† Water holding capacity
‡ Total Kjeldahl nitrogen

the RC facility controls the moisture content of compost so that it is relatively low at the end of the active composting process. The moisture content has to be in certain range for best recovery of screened compost. The pH values for the RC compost samples increased ($P<0.01$) and EC decreased ($p<0.01$) with compost curing time. These results are consistent with the findings of Iannotti et al. (1994) who reported similar results using municipal solid waste compost. Avnimelech (1996) also reported similar results and further explained that pH and EC changes were caused by decomposition of organic acids, suggesting that simple parameters such as pH and EC might be good indicators of compost stability for the purpose of compost process control. Eklind (1998) studied the effects of six different amendments on the composting processes and found in all cases that the compost pH increased from 6 to > 8 during the early stage of composting (<35 d) and then decreased slowly but steadily after that point. NH_4^+ is believed to be the cause of elevated pH in the early stage of composting (Avnimelech, 1996). These results suggest that the RC samples might be in a relatively early stage of curing. WPB samples showed consistent increase in pH ($p<0.01$) but the differences between samples were much smaller than RC samples, and only EC of WPB-3 was significantly ($p<0.01$) reduced with curing. This may indicate that the WPB samples were in a late curing stage. Since samples from the two facilities were actively composted and cured for about the same length of time, the differences between the two may be mainly due to the source waste materials and the efficiency of different composting systems. The compost samples from the SA facility exhibited very different chemical and physical properties when compared to the samples from the other two facilities. In general, they had the highest moisture contents and water holding capacities among the three compost groups

probably because of a high content of sawdust. In addition, they showed no consistent changes in pH and EC during the curing process. However, pH of SA-5 showed significant reduction ($p<0.01$), indicating that samples from SA facility excluding SA-5 had probably not been in an apparent stabilization and maturation stage. This conclusion is partly supported by the fact that the SA facility compost had a much higher percentage of TVS and a higher C/N ratio as well as lower concentrations of TKN and total P than the composts from the other two facilities. The changes in TVS % and C/N ratios during curing in compost from the three facilities failed to follow a consistent trend. The same was true for the concentrations of TKN and total P. A more thorough examination of these parameters, involving a long-term monitoring study of a single compost pile, found some correlation between compost curing time and these parameters (Adani et al., 1995; Chefetz, et al., 1998b). However, the results of the current tests indicate that, for a full scale composting facility with variations in waste feedstock and process controls, and limited composting time, these parameters are not suitable as accurate indicators for compost stability and maturity.

CO_2 Evolution

The CO_2 evolution rate decreased with curing time for the RC and WPB facility samples, and there was no clear trend in the CO_2 evolution rate for the SA samples (Fig 3-1). Due to the fluctuation of CO_2 evolution rate, the accumulated CO_2 evolution over 6-7 d of incubation was calculated (Table 3-2). The accumulated CO_2 evolution of RC samples consistently and significantly ($p<0.01$) decreased over time. WPB-2 and WPB-3 were significantly ($p<0.01$) lower than WPB-1. Only SA-5 sample was significantly lower ($p<0.01$) than the others from the same facility. This result corresponds well with the change of pH and EC. This further confirmed that RC samples were in a stabilization

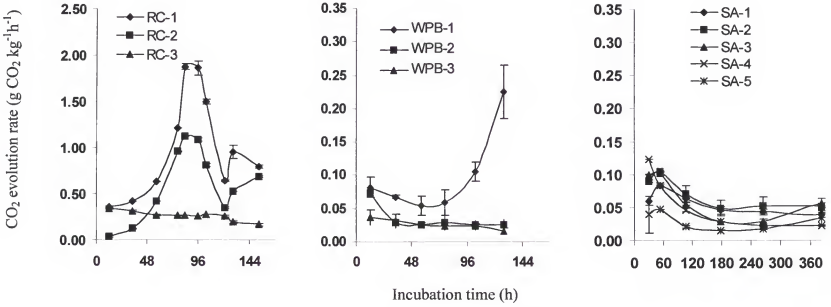

Figure 3-1. CO_2 evolution rate of compost samples with incubation time at 60% moisture content at 24±2 °C. Error bars stand for the standard deviation of three replicates of one composite sample in the lab.

Table 3-2. Cumulated CO_2 evolution after 7-8 d of incubation and germination rate after 3d.

Sample	CO_2 evolution ($g\ kg^{-1}$)	Germination rate (%)
RC-1	137.0 ± 1.0†	7 ± 12
RC-2	85.0 ± 4.1	41 ± 18
RC-3	38.6 ± 0.6	79 ± 10
WPB-1	20.4 ± 1.9	90 ± 10
WPB-2	3.7 ± 0.8	97 ± 4
WPB-3	4.5 ± 0.6	95 ± 8
SA-1	10.5 ± 0.3	52 ± 3
SA-2	12.5 ± 1.6	55 ± 13
SA-3	10.4 ± 0.2	31 ± 8
SA-4	10.1 ± 0.1	48 ± 5
SA-5	4.5 ± 0.7	41± 8

† Mean of three replicates ± standard deviation.

process, WPB samples were in a late curing stage, whereas most SA samples were not in an obvious stabilization process. Overall, the RC samples were significantly ($p<0.01$) higher in microbial activity than other two groups of samples. Based on CO_2 evolution, all the WPB and SA samples were much more stable than RC samples. There were much greater differences in values among three composting facilities than the differences within each composting facility imply that compost source materials and process have a great impact on the compost stabilization process.

Seed Germination

Samples from the RC facility exhibited a significant decrease ($p<0.01$) of phytotoxicity on tomato seeds with curing time (Table 3-2). All of the WPB samples yielded high germination rates (90-97%) which corresponded well to their overall low CO_2 evolution rates (Figure 3-1, Table 3-2). In contrast, the germination rates for the SA samples were all low (31-55%), indicating that the SA samples were still phytotoxic to tomato seeds despite their low CO_2 evolution. The EC and pH values for these samples were within the range that normally does not adversely affect seed germination. In addition, total trace metal concentrations and water soluble heavy metal concentrations were all low (data not shown). It can, therefore, be assumed that the low seed germination rate of the SA compost was associated with the feedstock source material and their actual composting stage (Zucconi, et al., 1981). These results demonstrated that stability based on CO_2 evolution and phytotoxicity based on seed germination tested different properties of compost, i.e. stability and maturity, and low respiration rates did not necessarily reflect low phytotoxicity.

Water Soluble Organic Carbon

Total WSOC concentrations in compost samples from three facilities decreased with curing time (Table 3-3), which is consistent with the literature (Chefetz et al., 1998a; Garcia et al., 1991a,b; Inbar et al., 1993). WSOC reduction is apparent in early stage of composting stage and gradually reaches a plateau with curing time (Chefetz et al., 1998c). The overall WSOC level of composts from the three facilities follows the order of RC>SA>WPB with significant difference ($p<0.01$) among facilities. Bernal et al. (1998b) established a WSOC value of <17 g kg^{-1} as the index for stable compost based on seven composts, whereas Hue and Liu (1995) used WSOC $\leq$ 10 g kg^{-1} as the cut-off point based on 17 composts. Using WSOC standards of 10-17 g kg^{-1} as the cut-off values for stable compost, the WPB and SA composts were both stable although the latter started out slightly higher than the standard (Table 3-3). These results are well correlated with accumulated CO_2 evolution ($R^2=0.781$, $p<0.01$, $n=11$), which supports the idea that WSOC is the most active part of carbon and indicative of compost stability. However, WSOC concentrations alone do not explain the low seed germination rate for the samples, especially when the data were compared to that of the RC-3 sample. These results suggest that while WSOC reduction can generally be correlated with reduced phytotoxicity to seed, it is important to consider the characteristics of WSOC composition. WSOC composition and phytotoxicity will change with different feedstock, bulking agents and other types of waste materials used along with compost curing time. Using MSA of WSOC, we may be able to indicate the phytotoxic property of WSOC. MSA is defined as the absorbance per mass unit of WSOC. A high MSA value for WSOC indicates a high degree of humification. MSA has previously been used to characterize the degree of humification of water-soluble humic substances (Battin,

Table 3-3. Concentration of dissolved organic carbon (WSOC), humic acid (HA) and fulvic acid plus nonhumic substance (FA) fraction and their mass specific absorbance (MSA) at 420 nm.

Sample	Curing (d)	Organic carbon concentration			Ratio of HA/FA	Mass Specific Absorbance ‡		
		WSOC†	HA †	FA †		WSOC†	HA †	FA †
		----------(g kg^{-1})----------				----------Lmg^{-1}m^{-1}----------		
RC-1	0	38.2 ± 0.14	4.12 ± 0.41	34.1 ± 0.57	0.12	0.12 ± 0.00	0.44 ± 0.04	0.05 ± 0.00
RC-2	7	29.8 ± 1.91	5.73 ± 0.01	24.1 ± 1.21	0.24	0.29 ± 0.02	0.67 ± 0.00	0.09 ± 0.00
RC-3	30	23.5 ± 0.17	8.18 ± 0.08	15.3 ± 0.03	0.53	0.33 ± 0.01	0.91 ± 0.01	0.12 ± 0.00
WPB-1	0	12.2 ± 0.70	2.69 ± 0.16	5.33 ± 0.08	0.50	0.26 ± 0.01	0.31 ± 0.02	0.11 ± 0.00
WPB-2	7	7.87 ± 0.27	2.82 ± 0.06	5.05 ± 0.08	0.56	0.27 ± 0.02	0.39 ± 0.01	0.11 ± 0.00
WPB-3	30	5.26 ± 0.29	2.45 ± 0.02	2.81 ± 0.03	0.87	0.44 ± 0.01	0.52 ± 0.02	0.17 ± 0.00
SA-1	0	20.5 ± 0.73	12.7 ± 0.09	7.88 ± 0.81	1.31	0.22 ± 0.01	0.29 ± 0.00	0.12 ±0.01
SA-2	7	19.0 ± 0.12	11.9 ± 0.10	7.10 ± 0.22	1.36	0.21 ± 0.01	0.26 ± 0.01	0.13 ± 0.00
SA-3	14	19.1 ± 0.53	11.9 ± 0.26	7.18 ± 0.27	1.46	0.22 ± 0.00	0.27 ± 0.00	0.13 ± 0.00
SA-4	16	17.5 ± 0.12	11.5 ± 0.13	6.04 ± 0.01	1.92	0.17 ± 0.01	0.20 ± 0.01	0.14 ± 0.00
SA-5	35	15.0 ± 1.22	8.62 ± 0.16	6.40 ± 1.06	1.01	0.18 ± 0.01	0.22 ± 0.00	0.10 ± 0.02

† Means of three replicates ± standard deviation.

‡ MSA is defined as absorbance at 420 nm per unit of mass of WSOC

1998). As expected, the MSA values for the HA fractions (1.9-9.1) were much greater than for the FA fractions (0.5-1.4, Table 3-3). As curing time increased, significant increases ($p<0.01$) in the MSA of WSOC, HA and FA were observed in the RC and WPB samples (Table 3-3). This implies that aromatic molecules were formed or concentrated during curing in these two composts. Mathur et al. (1993b) reported an increase followed by a decrease in WSOC concentration and absorbance with curing. Such a difference may be explained in two ways. First, Mathur et al. (1993b) did not express WSOC absorbance on a unit mass basis. Absorbance may decrease simply due to a decrease in total WSOC concentration. Second, the compost used by Mathur et al. (1993b) was in a final curing stage and was more stable than the compost samples used in our research. As such, much of the water soluble HA may have coagulated into a water-insoluble form (Schnitzer et al., 1993). This second reason may also explain the reduction of the HA fraction for the WPB samples. The advantage of using MSA can be seen by comparing the WSOC concentrations and MSA values for the HA fractions of composts from three facilities in our test. (Table 3-3). Based solely on the HA concentrations, the compost samples from the SA facility would be considered more mature. However, this is not the case upon further examination of the data. While the SA samples had the highest HA concentrations, their HA fractions had the lowest MSA, indicating that most of the organic matter in the HA fraction may not actually be humic acids. The SA facility used large amounts of sawdust as compost carbon source, its lignin component may have contributed to the HA fraction. As such, the use of MSA provides a better indication of the humification degree of WSOC than the carbon concentration ratio of HA/FA. Despite the variation caused by different waste material, seed germination rate was

negatively correlated to WSOC concentration (R^2=0.593, $p<0.01$, n=11) and positively correlated to the MSA of WSOC (R^2 =0.596, p=0.01, n=11). The results indicated that MSA of WSOC can serve as a simple but comprehensive index of WSOC composition for phytotoxicity when detailed analyses of chemical compounds in a compost are difficult. As a chemical method, WSOC analysis is much less time consuming and easier to standardize, without problems inherent to biological tests. More data are required to confirm the relation between compost WSOC and respiration rate as well as the phytotoxicity of biosolids compost, nevertheless it is promising that WSOC and MSA can be used to assess compost stability and maturity status.

Summary

Compost stability based on CO_2 evolution and compost maturity based on seed germination are indeed two different characteristics of compost quality. Generally, stability and maturity may be correlated, e.g. more stable compost tends to be more mature. However, due to variation in compost feedstock and composting processes, some 'stable' compost may need more time to break down the phytotoxic substances whereas 'mature' compost may have a relatively high respiration rate. As a result, both parameters are needed to assure quality compost product. WSOC concentration and MSA are highly correlated to CO_2 evolution and germination rate. WSOC analysis can potentially replace the CO_2 evolution and seed germination test to assess compost stability and maturity. In a full scale composting facility, C/N ratio and HA /FA of WSOC are not accurate indicators of compost stabilization and maturation processes. Instead, with relatively consistent source waste composition and calibration of other

stability and maturity tests, pH and EC may be used to monitor compost stabilization and maturation processes.

CHAPTER 4
EFFECTS OF SAMPLE STORAGE ON BIOSOLID COMPOST STABILITY AND MATURITY EVALUATION

Introduction

For most studies conducted to evaluate compost stability and maturity (Bernal et al., 1998b; Henry and Harrison, 1996; Mathur et al., 1993a), fresh compost samples with minimum storage time have been used since sample storage may affect compost stability and maturity. However, in reality, sampling and analysis cannot always be carried out simultaneously. As a result, there is a need to study the effects of sample storage on compost stability and maturity, and develop a satisfactory method to preserve compost samples for stability and maturity evaluation.

To date, there has been little work done to study the storage effect on compost stability and maturity. However, relevant information is available from study of storage effects on soil analyses. Proper storage method depends on the parameters to be analyzed. For chemical analysis, air drying of soil samples is the most common practice (Bates, 1993), despite the concern that drying and rewetting of soil may significantly alter the results of some chemical analyses (Rayment, 1993; Slattery and Burnett, 1992), especially analyses involving soil solution chemistry (Walworth, 1992). For microbial analysis, refrigeration and freezing moist soil are the most commonly used methods (Stenberg, et al., 1998). On the other hand, air-dry samples are considered least suitable because the air drying process alters microbial metabolism and reduces the diversity of microbial species (Zelles et al., 1991). Refrigeration of samples at 2-4 °C is commonly

recommended for analysis on soils with short-term storage of less than three months (Brohon et al., 1999). However, slow depletion of available substrate in soil samples is expected due to the ongoing microbial activity (Coxson and Parkinson, 1987) that occurs even with refrigeration. The impact of freezing on microbial activity is not as well defined. Freezing reduces microbial activity by cell lysis during ice formation (MacLeod and Calcott, 1976). However, the freezing and subsequent thawing of soil can also cause improved aggregate dispersal (Winter et al., 1994). This may reduce the physical protection of organic matter and microbial biomass thus enhance microbial activity (Hassink 1995). The impacts of frozen storage on sample analysis depend on the types of soil samples used and microbial parameters analyzed. Winter et al. (1994) discouraged the use of sample freezing for microbial biomass C analysis. Although no significant detrimental effects were actually observed, they believe that the impacts of freezing are masked by improvements in extraction efficiency with frozen samples. Stenberg et al. (1998) concluded that storage at –20 °C for 13 months had no effect on the microflora in annually frozen soils. The US Composting Council (1998) recommended 4° C for short term storage of less then 2 weeks, and –4°C for long term storage of compost samples for biological analysis, including compost stability and maturity evaluation. However, the maximum time that samples can be stored under this condition was not specified.

Compost stability and maturity are comprehensive properties indicating the degree of organic matter decomposition and potential of phytotoxicity caused by incomplete composting. Since composting is a microbial process, compost stability and maturity are greatly impacted by microbial activity. We hypothesize that the chemical composition and organic matter decomposition status of compost will play an important

role in determining microbial activity. During storage, if microbial activity is minimal, compost stability and maturity status should remain unchanged unless there is significant disruption of the physical and chemical properties of compost samples. As a result, preserved compost samples can, in principle, be used for compost stability and maturity tests.

Except for extremely mature compost, most compost has a relatively high organic matter content with potentially available organic carbon and nutrients, which will support relatively high microbial population or activity. Thus, storing compost at 2-4 °C for a long time may significantly change its compost chemistry, especially for the less stable or less mature composts, due to the ongoing microbial activity. It is desirable to completely stop all microbial activity during long-term storage. We saw visible fungal colonies in samples stored for 2-3 month at 4°C, which indicated that refrigeration was not a suitable method for storing sample for up to a year. As a result, refrigeration storage was not chosen for this study. Rather air-drying and freezing storage methods were selected to minimize the microbial activity during the long period of sample storage.

The objective of this study was to compare the effects of the two sample storage methods on compost stability and maturity. Compost samples were stored at room temperature (24±2°C) after air drying or were stored frozen (-18 to -20°C) for a period of 12 months. Test results from stored compost samples using these methods were compared to those of fresh compost samples. Three methods of evaluating compost stability and maturity were tested: the respiration activity test based on CO_2 evolution, the seed germination rate test for phytotoxicity and a water-soluble organic carbon (WSOC) test.

Materials and Methods

Compost Sample Preparation and Characterization

Compost samples used in this part of study included all RC and WPB samples and three samples from SA. They were SA-1, SA-3 and SA-5. In addition, two MW samples from a fourth biosolid composting facility were used. Compost samples were collected and screened as described in chapter 2 and 3. A portion of the screened samples was kept refrigerated for less than a week before being used. A subsample was dried at 45°C and stored in a sealed plastic bag at room temperature. Another subsample was sealed in a plastic bag. Plastic bags containing samples were randomly placed in a freezer at –20 °C. Since the refrigeration storage was less than a week, these samples were considered "fresh" compared with the air-dried and frozen samples, which were subsequently stored for one year. Three parameters of compost stability and maturity, i.e., CO_2 evolution, phytotoxicity and WSOC, were performed on both the "fresh" and stored samples. The physical and chemical characteristics of samples are listed in Table 3-1 and Table 4-1. Detailed analytical methods for physical and chemical characterization were described in Chapter 3.

Compost Stability and Maturity Evaluation

The procedure of measuring the three parameters of compost stability and maturity, i.e., CO_2 evolution, phytotoxicity and WSOC, were described in Chapter 3. Peak CO_2 evolution rate was obtained by averaging the highest CO_2 evolution rate points of the three replicates for each compost sample.

Table 4-1. Selected physical and chemical properties of tested biosolids compost samples.

Sample	Curing (d)	Moisture content ($g\ kg^{-1}$)	WHC† ($g\ kg^{-1}$)	pH (water)	Electrical conductivity ($S\ m^{-1}$)	Volatile solids ($g\ kg^{-1}$)	TKN ‡ ($g\ kg^{-1}$)	Organic Carbon ($g\ kg^{-1}$)	C/N ratio
MW-1	30	527	669	6.4	0.13	221	11.3	124	11
MW-2	90	435	473	6.0	0.09	164	6.9	90	13

† Water holding capacity
‡ Total Kjeldahl nitrogen

Statistical Analyses

The SAS program was used to detect statistically significant differences ($P<0.05$) between treatments (SAS, 1987). Effects of two main factors: samples and storage methods, were tested using a GLM procedure. Duncan's multiples range test was used to calculate significant difference among storage treatments for each sample. Correlation coefficients were calculated among the three parameters using a CORR procedure.

Results and Discussion

CO_2 Evolution Rate

The CO_2 evolution rate fluctuated during the incubation (Fig 4-1). Less stable compost exhibited greater variations over time and formed obvious peaks of CO_2 evolution (RC-1 and RC-2 samples). Peaking of CO_2 evolution has been previously reported (Hue and Liu, 1995; Iannotti et al., 1994, Lasaridi and Stentiford, 1998) and the time it takes to form CO_2 evolution peaks varied greatly among different samples. As a result, the average CO_2 evolution or the total CO_2 evolution for 3 d has often been used to represent compost stability. However, the 3-d cutoff could not be used in the present study because the time to reach the maximum CO_2 activity was affected by the storage method for some samples. For example, the fresh RC-1 sample took about 3 to 4 d to reach its CO_2 evolution peak while the air-dried and frozen samples took about 4 to 5 and 7 to 9 d, respectively (Fig 4-1). Samples that took longer time to reach peak CO_2 evolution, such as frozen RC-1 (Table 4-2, 4-3), will inevitably have lower accumulated CO_2 evolution, thus providing misleading results. For more stable compost samples, the overall CO_2 evolution rate was much lower and exhibited no clear trend of peaking (Fig. 4-1). This implies that there is no advantage in using peak CO_2 evolution versus

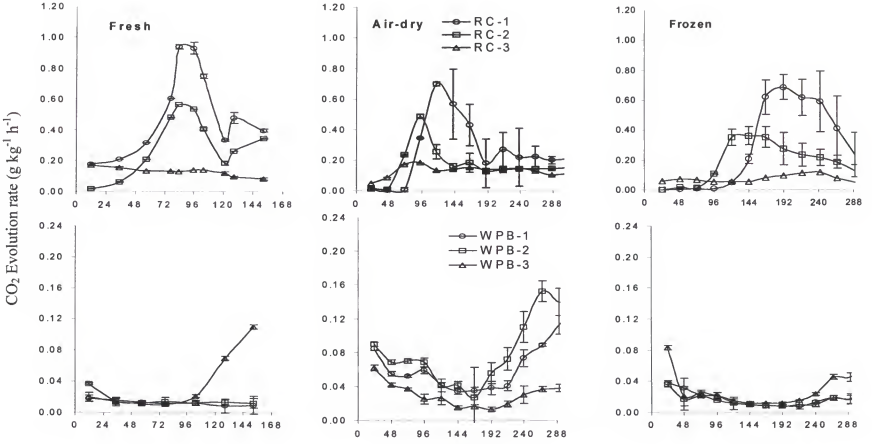

Figure 4-1. CO_2 evolution rate of fresh, air-dried and frozen samples over the incubation time. The first column is the fresh samples, the middle one is the air-dried samples and last one is the frozen samples. The error bars represent standard deviation of three replicates.

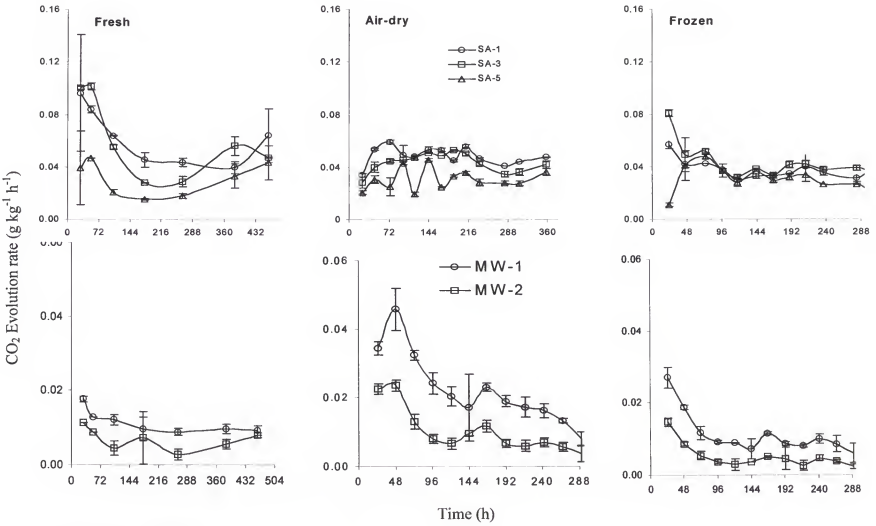

Figure 4-1. Continued.

Table 4-2. Peak CO_2 evolution rates of fresh, air-dry and frozen compost samples†.

Sample	Fresh	Air-dry	Frozen
	g CO_2 kg^{-1} dm h^{-1}		
RC-1	0.96 ± 0.02a‡	0.77 ± 0.03b	0.74 ±0.03c
RC-2	0.48 ± 0.00a	0.49 ± 0.01a	0.42 ±0.06b
RC-3	0.17 ± 0.00a	0.18 ± 0.01a	0.12 ±0.01b
WPB-1	0.11 ± 0.01a	0.08 ± 0.00a	0.04 ±0.00b
WPB-2	0.02 ± 0.00b	0.09 ± 0.00a	0.04 ±0.00b
WPB-3	0.04 ± 0.01b	0.06 ± 0.00ab	0.08 ±0.00a
SA-1	0.09 ± 0.00a	0.06 ± 0.00a	0.06 ±0.00a
SA-3	0.10 ± 0.00a	0.05 ± 0.00b	0.07 ±0.02a
SA-5	0.05 ± 0.00a	0.05 ± 0.01a	0.05 ±0.00a
MW-1	0.02 ± 0.00a	0.05 ± 0.01a	0.03 ±0.00a
MW-2	0.01 ± 0.00a	0.02 ± 0.00a	0.01 ±0.00a
Overall Means	0.20 ± 0.29a	0.18 ± 0.24b	0.16 ±0.23c

† Measurements were taken at 24±2°C and 60% of moisture content in the lab during a period of incubation for <14 days.

‡ One standard deviation of 3 replicates, means of different treatments on each sample (same row) that are followed by the same letter are not significantly different (α=0.05) using Duncan's Multiple Range Test.

Table 4-3. Cumulated CO_2 evolution of fresh, air-dry and frozen compost samples after 7 days†.

Sample	Fresh	air-dry	Frozen
		g CO_2 kg^{-1} dm	
RC-1	136.49 ± 0.92a	49.17 ± 3.23b	0.51 ± 0.51c
RC-2	85.00 ± 4.11a	31.90 ± 1.05c	37.03 ± 0.71b
RC-3	38.57 ± 0.64a	21.93 ± 0.51b	14.45 ± 0.10c
WPB-1	11.90 ± 0.73a	8.69 ± 0.36b	3.47 ± 0.20c
WPB-2	4.52 ± 0.64b	9.74 ± 0.63a	2.82 ± 0.15c
WPB-3	3.68 ± 0.78a	5.38 ± 0.24a	4.56 ± 0.50a
SA-1	10.47 ± 0.25a	8.46 ± 0.21b	6.60 ± 0.64b
SA-3	10.38 ± 0.19a	7.33 ± 0.26b	7.78 ± 0.52b
SA-5	4.49 ± 0.78a	4.42 ± 0.06a	5.62 ± 0.12a
MW-1	2.17 ± 0.18b	4.72 ± 0.39a	2.28 ± 0.05b
MW-2	1.31 ± 0.61a	2.28 ± 0.21a	1.06 ± 0.05a
Overall Means	28.14 ± 43.81 a	14.06 ± 14.59b	7.87 ± 10.41c

† Measurements were taken at 24±2°C and 60% of moisture content in the lab during a period of incubation for 14 days.

‡ One standard deviation of 3 replicates, means of different treatments on each sample (same row) that are followed by the same letter are not significantly different (α=0.05) using Duncan's Multiple Range Test.

accumulated CO_2 evolution for more stable compost. In other words, for more stable compost, either method will provide valid data for evaluating compost stability. The delay in CO_2 evolution peaking and different shapes of CO_2 evolution in the active samples are probably the result of changes in microbiology such as microbial population or activities of the stored sample. The difference between active and stable sample is probably because the more active samples have higher numbers of active microbes which are sensitive to freezing and air-drying, than the more stable samples (MacLeod and Calcott, 1976).

Seed Germination

With the exception of the SA samples, no significant storage effect was noted in the seed germination test for the compost samples. Surprisingly, both air drying and freezing storage significantly ($p<0.01$) increased seed germination of the SA compost samples (Table 4-4). Many substances found in immature compost can lead to a reduction in seed germination rate with its magnitude depending on the source waste material and composting process. For example, a variety of organic compounds including short- (Shiralipour et al., 1997) and long-chain fatty acids (Sesay et al., 1997) and phenolic acids (Leviminzi et al., 1994; Ishii and Kadoya, 1993; Janovicek et al., 1997) inhibit seed germination. However, many of these substances could coexist in immature compost. It has been found that a combination of volatile acids in an extract of an immature compost was phytotoxic to lettuce seedlings at concentrations far lower than the minimum levels at which individual acids such as formic, acetic, benzoic, salicylic and tannic acids exerted any deleterious effects (Manios, et. al. 1987). The SA samples were very fibrous, containing large amounts of sawdust, and were generally different

Table 4-4. Seed germination rates measured in fresh, air-dry and frozen stored samples.

Sample	Fresh	Air-dry	Frozen
	% of water as control		
RC-1	7 ± 12†a	9 ± 2a	2 ± 2a
RC-2	41 ± 18b	65 ± 16a	46 ± 20ab
RC-3	79 ± 10a	79 ± 17a	82 ± 0a
WPB-1	90 ± 10a	90 ± 7a	63 ± 13a
WPB-2	97 ± 4a	90 ± 6a	88 ± 9a
WPB-3	95 ± 8a	89 ± 4a	96 ± 3a
SA-1	52 ± 6b	89 ± 9a	82 ± 9a
SA-3	31 ± 8b	84 ± 11a	87 ± 6a
SA-5	41 ± 8b	86 ± 12a	72 ± 8a
MW-1	89 ± 9a	85 ± 10a	88 ± 10a
MW-2	87 ± 5a	89 ± 12a	89 ± 8a
Overall Means	66 ± 31c	77 ± 23c	72 ± 27b

† One standard deviation of 3 replicates, means of different treatments on each sample (same row) that are followed by the same letter are not significantly different (α=0.05) using Duncan's Multiple Range Test.

physically and chemically from the other samples. Therefore the increase in seed germination for the SA samples is probably related to the source material or its composting process. On the other hand, the RC samples, which was the only group that showed a clear trend of maturation with curing, exhibited consistent and comparable seed germination inhibition among the different storage treatments (Table 4-4). The results from the RC sample group demonstrate that proper storage does not significantly alter the phytotoxic properties of immature compost.

Water Soluble Organic Carbon

The WSOC was more affected by the storage treatments than CO_2 evolution and seed germination test. Except for the most stable MW samples, air-dry storage significantly reduced the amount of WSOC, whereas frozen storage significantly increased the WSOC concentration (Table 4-5). The reduction in WSOC for the air-dried samples could be due to loss of volatile organic substances. The freezing-thawing process has been reported to enhance the water extractability of soil organic carbon due to disruptions in the physical structure of soil samples and release of microbial cell contents as a result of cell wall rupture by ice formation (Winter et al., 1994).

Relation of WSOC to CO_2 Evolution and Seed Germination

Despite the inconsistency of storage effects on the three measured parameters, there was high correlation between the parameters. Peak CO_2 evolution rate was exponentially related to WSOC with R^2 =0.92 (<0.01, n=11), 0.81 (<0.01, n=11), and 0.83 (<0.01, n=11) for the fresh, air-dried and frozen samples, respectively (Figure 4-2). The coefficient of determination between CO_2 evolution rate and WSOC for all samples was 0.78 (<0.01, n=33). WSOC was also linearly correlated with the seed germination

Table 4-5. Water soluble organic carbon (WSOC) concentration of freshly collected, air-dry and frozen stored samples.

Sample	Fresh	Air-dry	Frozen
	g kg^{-1} dry matter		
RC-1	38.2 ± 0.1a†	29.7 ± 0.3b	38.6 ±0.3a
RC-2	29.8 ± 1.9b	27.2 ± 1.8c	35.2 ± 0.1a
RC-3	23.5 ± 0.2b	19.2 ± 1.9c	25.3 ± 0.1a
WPB-1	8.1 ± 0.7b	6.8 ± 0.2c	9.7 ± 0.3a
WPB-2	7.9 ± 0.3b	6.5 ± 0.7c	9.2 ± 0.3a
WPB-3	5.3 ± 0.3b	3.5 ± 0.1c	6.5 ± 0.2a
SA-1	20.5 ± 0.7b	13.3 ± 0.8c	21.7 ± 1.1a
SA-3	19.1 ± 0.5b	13.2 ± 1.6c	23.0 ± 12a
SA-5	15.0 ± 0.2b	10.5 ± 0.0c	14.4 ± 1.0a
MW-1	0.8 ± 0.0a	0.6 ± 0.5a	0.7 ± 0.2a
MW-2	0.3 ± 0.0a	0.4 ±0.1a	0.3 ± 0.1a
Overall Means	15.7 ± 12.0c	11.9 ± 10.0b	16.8 ± 13.0a

† One standard deviation of 3 replicates, means of different treatments on each sample (same row) that are followed by the same letter are not significantly different (α=0.05) using Duncan's Multiple Range Test.

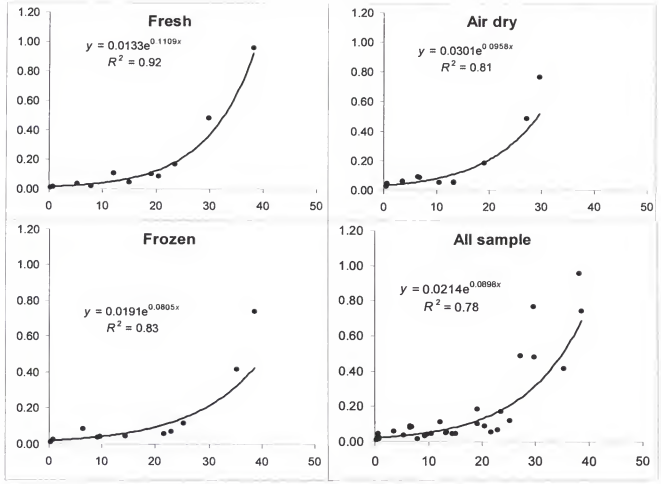

Figure 4-2. Relation between compost water soluble organic carbon and peak CO_2 evolution rate.

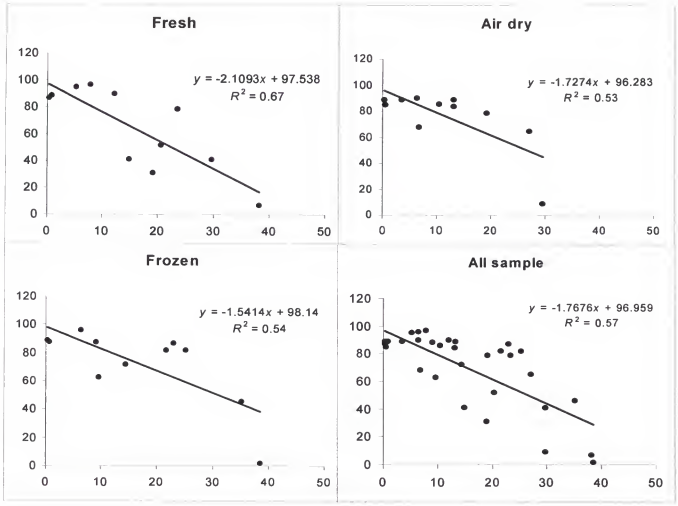

Figure 4-3. Relation between compost water soluble organic carbon and tomato seed germination.

rate. The coefficient of determination between WSOC and seed germination rate was 0.67 (<0.01, n=11), 0.53 (<0.01, n=11) and 0.54 (<0.01 n=11) for fresh, air-dry and frozen samples, respectively, and that of all samples was 0.57 (<0.01, n=33) (Fig. 4-3). Similar and significant correlation among different storage methods indicates that consistent relationships exist between WSOC, CO_2 evolution and seed germination, and that the relationships are not seriously impacted by sample storage.

Summary

Depending on the stability and maturity level of the compost and its feedstock source, air-drying and freezing samples had varied effects on the three measured parameters: CO_2 evolution, seed germination, and WSOC concentration. Neither of the two storage methods is satisfactory in terms of maintaining the original properties of the fresh compost. Storage tends to delay the peaking of CO_2 evolution rate of the active sample, causing much lower accumulated CO_2 evolution during a fixed period of time. Peak CO_2 evolution rate, rather than average or total CO_2 evolution over time, was thus a more suitable parameter for evaluating compost stability for compost of high microbial activity. It is possible to preserve compost samples for phytotoxicity analysis. However, fresh samples without storage is recommended for phytotoxicity test until the storage effect is further understood. The WSOC concentrations decreased with air-dry storage and increased with frozen storage. The storage effect was not significant on compost with low initial WSOC. Despite all these variations, WSOC has a significant and consistent correlation with both CO_2 evolution and seed germination.

CHAPTER 5
RELATION BETWEEN COMPOST STABILITY AND EXTRACTABLE ORGANIC CARBON

Introduction

Humification is widely considered as an important process during composting of organic materials where humic substances form and non-humic substances decompose (Bernal et al., 1998b; Hsu and Lo 1999; Leita and Denobili, 1991; Miikki, et al., 1997; Sanchez-Monedero et al., 1999; Schnitzer et al., 1993). As composting progresses, the percentage of humic substance is expected to increase relative to the total dry mass or the total organic matter. As a result, humification-related parameters have been examined to represent compost stability and maturity (Adani et al., 1997; Garcia et al., 1991a; Jimenez and Garcia, 1992; Veeken et al., 2000;).

The method commonly used to extract humic substances from composts is similar to those used to extract organic matter from soils, where humic substances are extracted with dilute sodium hydroxide or sodium pyrophosphate solution, or a mixture of the two. The fraction that precipitates at pH<2 is referred to as humic acid (HA), whereas the fraction remaining soluble at pH<2 contains fulvic acid and non-humic substances (FA). Contrary to what is expected, concentrations of HA may decrease during composting or the differences in HA concentrations between raw material and mature compost may not be significant (Bernal et al., 1996; Garcia et al., 1991a; Jimenez and Garcia, 1992). This discrepancy may be due to the fact that NaOH-extractable organic carbon (OC) contains

a considerable amount of biodegradable non-humus fraction, especially during the initial stages of composting (Adani, 1995).

Chefetz et. al. (1998a) developed the concept of core-HA fraction by removing the non-humic substance from the HA fraction using organic solvents and sulfuric acid extractions prior to the alkaline extraction, a process believed not to alter the core-HA fraction. They found that the OC content in core-HA relative to total dry mass increased while HA decreased steadily with composting. Adani (1995) found that the difference in OC between HA and core-HA fractions decreased during composting and proposed to use the ratio of core-HA/HA as a new compost stability index (organic matter evolution index, OMEI), with a maximum level approaching to 1. Though the relation of core-HA to HA casts insight into OC evolution during composting process, it cannot be used as a practical indicator for compost stability. This is because the measurement of this index involves extensive laboratory work.

In addition to the ratio of core-HA/HA, the ratio of HA/FA has been used to assess organic matter stability. It is found that the ratio of HA/FA invariably increased during composting, especially when the fraction of NaOH-soluble OC at pH<2 is treated as FA (Garcia 1991a). This is because that the FA fraction decreased more than the HA fraction during composting. However, due to the great variability among composts of different material sources, the suggested critical value of the index spans a large range for different composts, even within the same composting system and time (Bernal et al., 1998b). As a result, it is not feasible to use the single index of HA/FA to determine compost stability.

The primary objectives of this study were to 1) investigate the extractability of organic carbon from compost of different source and stability, and 2) to exam the relation between water- and NaOH-extractable OC, and compost stability estimated by the CO_2 evolution. The extracted OC was further separated into FA and HA fractions to assess their relations with compost stability. The ultimate goal of this research was to identify a simple and reliable way to estimate compost stability for composts from different source materials.

Materials and Methods

Compost Samples

All of the biosolids compost samples listed in Table 2-1 except SA-2 and SA-4 was used for this study. The Puerto Rico (PR) samples were collected from Puerto Rico and were freeze-dried before shipping to our lab. For other composts, air-dried samples were used for the analysis of CO_2 evolution and sequential extraction of organic carbon. The selected chemical and physical properties of compost samples RC2 and PR are listed in Table 5-1. For the rest samples, see Table 3-1. For details on measurement of these parameters, see Chapter 3.

Sequential Extraction and Analysis of Organic Carbon

A three-step sequential extraction procedure was employed to separate compost OC into different fractions. Samples were first extracted with water for 2 h (water-2h), then with 0.1 N NaOH solution for 2 h (NaOH-2h), and finally 0.1N NaOH solution for 24 h (NaOH-24h), with the solid to solution ratio being 1:10. The extraction was conducted in a horizontal shaker at room temperature under N_2 atmosphere. At the end

Table 5-1. Select physical and chemical properties of tested biosolids compost samples.

Sample	Curing (d)	WHC† ($g\ kg^{-1}$)	Volatile solids ($g\ kg^{-1}$)	Total N ($g\ kg^{-1}$)	OC ($g\ kg^{-1}$)	C/N ratio
RC2-1	0	1376	852	43.0	639	15
RC2-2	9	1415	884	41.0	663	16
RC2-3	21	1169	681	36.0	511	14
RC2-4	25	1127	646	37.0	485	13
PR-1	0	716	358	13.6	203	15
PR-2	7	699	324	12.2	183	15
PR-3	14	662	297	12.6	168	13

† Water holding capacity.

of 2 or 24 h of shaking, the suspension was centrifuged at 9, 630 g for 10 min and filtered through a 0.45 μm membrane filter. The OC content and mass specific absorbence (MSA) of each fraction were determined according to the procedures as described in Chapter 2.

CO_2 Evolution

Compost stability was measured based on CO_2 evolution rate as described in Chapter 2 and the peak CO_2 evolution rate was used to represent compost stability.

Statistical Analyses

The SAS (release 6.12) procedure GLM with LSMEAN and PDIFF options was used to compute the p value of statistical differences between treatments. Samples from each facility were treated as nested by facility. Correlation coefficients between parameters were calculated using a CORR procedure. Stepwise multiple correlation procedure was used to derive the multiple regression between CO_2 evolution rates and other parameters.

Results and Discussion

Compost samples used in this study varied greatly in their physical and chemical properties (Table 2-1), which impacted compost stability measurement. In general, SA samples had the highest water holding capacity and C/N ratio as a result of their high content of volatile solids and OC, whereas RC samples had relatively high total N and OC. On the other hand, MW and PR samples had generally low values for all the parameters determined in this study. These differences among compost samples were mainly due to the differences in their source material and composting process.

CO_2 Evolution Rate

CO_2 evolution generally decreased with curing (Table 5-2). Decrease in CO_2 evolution rates was more significant ($p<0.01$) in the RC samples, which suggested these samples were not stable. In comparison, decreases in those of the remaining samples were only minor, indicating that they were relatively stable (Table 5-2). Hue et al. (1995) used the mean plus two standard deviations of CO_2 evolution rate as the cut-off value for stable compost, which is based on 14 commercial composts (presumably stable). The value they calculated is 120 mg CO_2 kg^{-1} dm hr^{-1} based on the average of the last 2 d of a 3-d incubation test. Based on the standard of 120 mg CO_2 kg^{-1} dm hr^{-1}, all compost samples in the current study were stable except for the five samples from RC facility with CO_2 evolution rate >120 mg CO_2 kg^{-1} dm hr^{-1} (Table 5-2). This was consistent with the conclusion derived based on changes in CO_2 evolution rate with curing. The average CO_2 evolution rate for the remaining samples was 55 mg CO_2 kg^{-1} dm hr^{-1} with a standard deviation of 21 mg CO_2 kg^{-1} dm hr^{-1}. A value of 98 mg CO_2 kg^{-1} dm hr^{-1} was obtained as the cut-off value based on this current study following the method of Hue et al. (1995), which was close to the reported120 mg CO_2 kg^{-1} dm hr^{-1}. This suggests that the index based on CO_2 evolution rate may be a good parameter to screen for unstable composts with diverse source materials.

Water- and NaOH-Extractable Organic Carbon

Organic carbon in composts was sequentially extracted with water and NaOH (three sequential extractions), which was further separated into HA and FA fractions for

Table 5-2. Peak CO_2 evolution rate (g CO_2 kg^{-1} dm hr^{-1}) and organic carbon content of sequentially extracted organic matter (g kg^{-1}, total dry mass).

Compost	Curing D	CO_2	Water				0.1 NaOH for 2 hr			
			HA	FA	Sum	HA/FA	HA	FA	Sum	HA/FA
RC-1	0	768 ± 29	3.6 ± 1.1†	25.2 ± 1.0	28.8 ± 0.1	0.14	23.1 ± 0.6	17.8 ± 0.1	40.9 ± 0.5	1.30
RC-1	7	487 ± 14	6.0 ± 1.4	20.0 ± 1.8	26.0 ± 0.4	0.30	19.0 ± 0.2	14.4 ± 0.5	33.4 ± 0.7	1.32
RC-1	30	185 ± 7	4.5 ± 0.1	14.2 ± 0.1	18.7 ± 0.1	0.31	19.8 ± 1.6	11.7 ± 0.8	31.4 ± 0.7	1.70
RC2	0	686 ± 15	3.7 ± 1.9	31.1 ± 0.0	34.7 ± 2.0	0.12	16.6 ± 0.4	19.3 ± 0.1	35.9 ± 0.3	0.86
RC2	9	606 ± 50	5.9 ± 0.2	35.5 ± 1.0	41.5 ± 1.2	0.17	16.5 ± 0.3	17.3 ± 0.3	33.8 ± 0.0	0.95
RC2	21	80 ± 9	9.3 ± 0.2	17.4 ± 0.6	26.7 ± 0.4	0.54	16.5 ± 0.4	9.6 ± 0.2	26.0 ± 0.5	1.72
RC2	25	17 ± 5	9.2 ± 0.5	9.4 ± 0.0	18.6 ± 0.4	0.99	23.1 ± 0.4	6.6 ± 0.1	29.8 ± 0.5	3.48
WPB	0	65 ± 2	2.5 ± 0.1	4.9 ± 0.1	7.4 ± 0.1	0.51	11.5 ± 0.2	10.6 ± 0.5	22.0 ± 0.3	1.09
WPB	7	90 ± 2	2.4 ± 0.2	5.0 ± 0.1	7.3 ± 0.2	0.48	7.9 ± 2.1	10.7 ± 0.1	18.6 ± 2.0	0.74
WPB	30	62 ± 3	1.4 ± 0.1	2.7 ± 0.0	4.1 ± 0.1	0.51	13.7 ± 1.1	6.6 ± 0.7	20.3 ± 0.3	2.11
SA	0	60 ± 2	3.4 ± 0.0	8.8 ± 0.3	12.2 ± 0.3	0.38	10.7 ± 1.8	7.9 ± 0.1	18.7 ± 1.8	1.35
SA	14	53 ± 1	2.9 ± 0.0	8.9 ± 0.2	11.8 ± 0.2	0.33	6.1 ± 1.1	8.2 ± 1.1	14.3 ± 0.0	0.75
SA	30	45 ± 4	2.2 ± 0.1	7.1 ± 0.1	9.3 ± 0.0	0.32	10.1 ± 0.5	7.3 ± 0.2	17.4 ± 0.3	1.39
MW	60	46 ± 6	0.4 ± 0.0	1.2 ± 0.0	1.6 ± 0.0	0.34	8.0 ± 0.5	6.1 ± 0.8	14.2 ± 0.3	1.32
MW	90	23 ± 2	0.1 ± 0.0	0.9 ± 0.1	1.0 ± 0.1	0.17	6.2 ± 0.7	5.9 ± 0.6	12.1 ± 0.1	1.07
PR	0	54 ± 2	0.7 ± 0.5	6.3 ± 0.5	6.9 ± 0.0	0.11	4.9 ± 0.3	8.4 ± 0.2	13.3 ± 0.4	0.58
PR	7	58 ± 5	0.6 ± 0.0	3.7 ± 0.0	4.3 ± 0.0	0.15	3.9 ± 0.3	7.3 ± 0.0	11.2 ± 0.3	0.54
PR	14	40 ± 1	0.8 ± 0.0	5.0 ± 0.1	5.9 ± 0.0	0.16	3.4 ± 0.1	8.2 ± 0.2	11.6 ± 0.1	0.41

† One standard deviation of 3 replicates.

Table 5-2--Continued.

Compo	Curing	0.1 NaOH for 24 hr				total
st	D	HA	FA	Sum	HA/FA	
RC-1	0	32.6 ± 1.1	15.1 ± 0.6	47.8 ± 0.5	2.16	117.4 ± 0.1
RC-1	7	26.5 ± 1.3	15.2 ± 0.8	41.7 ± 2.1	1.74	101.1± 3.2
RC-1	30	23.3 ± 0.0	11.7 ± 0.3	35.0 ± 0.3	2.00	85.1 ± 1.1
RC2	0	31.3 ± 0.3	16.1 ± 1.1	47.4 ± 0.9	1.95	118.0 ± 0.8
RC2	9	34.2 ± 6.8	14.0 ± 1.3	48.2 ± 5.5	2.47	123.6 ± 6.7
RC2	21	26.5 ± 1.5	9.2 ± 0.4	35.7 ± 1.9	2.87	88.5 ± 0.9
RC2	25	27.3 ± 1.1	6.9 ± 0.2	34.2 ± 1.3	3.94	82.6 ± 1.4
WPB	0	24.5 ± 1.7	13.0 ± 0.9	37.4 ± 0.8	1.90	66.8 ± 1.1
WPB	7	21.5 ± 0.1	11.7 ± .01	33.2 ± 0.0	1.84	59.1 ± 1.8
WPB	30	24.1 ± 0.2	6.0 ± 0.3	30.1 ± 0.1	4.01	54.5 ± 0.2
SA	0	10.5 ± 1.5	7.5 ± 0.2	18.0 ± 1.7	1.41	48.8 ± 0.5
SA	14	10.7 ± 2.6	7.6 ± 0.1	18.3 ± 2.7	1.42	44.4 ± 2.5
SA	30	11.2 ± 0.3	7.5 ± 0.5	18.7 ± 0.7	1.50	45.4 ± 0.1
MW	60	20.8 ± 1.3	12.8 ± 1.7	33.6 ± 3.0	1.63	49.4 ± 3.3
MW	90	21.3 ± 0.4	9.7 ± 1.9	31.0 ± 1.6	2.23	44.2 ± 1.7
PR	0	6.8 ± 0.8	5.9 ± 0.4	12.7 ± 1.3	1.14	32.9 ± 1.7
PR	7	6.2 ± 0.7	6.1 ± 0.7	12.4 ± 0.0	1.03	27.9 ± 0.3
PR	14	6.4 ± 0.6	5.8 ± 0.0	12.2 ± 0.6	1.11	29.7 ± 0.4

each extraction. In addition, the sum of HA and FA, and the ratio of HA/FA were also calculated. Thus, there were a total of 12 parameters based on various fractions of OC content. The peak CO_2 evolution rates of all compost samples, together with their corresponding OC contents, are presented in Table 5-2.

The total extractable OC content (sum of all three extractions) ranged from 27.9 to 123.6 g kg^{-1} dry mass, with the RC samples having the greatest concentration among all compost samples (Table 5-2). The total extractable OC was highly correlated (R^2= 0.881, $p<0.01$) with total volatile solids excluding the SA samples. The SA samples had a lower extractable OC than other samples, probably due to the high amount of sawdust added to the compost.

As expected, the distribution of extractable OC (sum of HA and FA) among the three extractions increased in the order of water-2h (2-34%), NaOH-2 h (27-40%) and NaOH-24 h (39-70%) as the extraction intensity increased, which was mainly contributed from a consistent increase in the HA fraction (Table 5-2). The increase in the HA fraction with increasing extraction intensity resulted in an increase in the ratio of HA/FA. It is generally believed that FA is more soluble, thus more easily extractable than HA.

It is reasonable to assume that the water-extractable OC would be more easily degradable than the NaOH-extractable OC, which was confirmed by the data. With curing, the reduction in the water-extractable OC decreased more than that in the NaOH-extractable OC (Table 5-2). For example RC-1 and RC2 samples, reduction in total water-extractable OC with curing was 39% and 46% compared to <26% and <28% for the NaOH-extractable OC. This is partially because the greater ratios of HA/FA in the NaOH-extractable OC than those in the water-extractable OC. However, no consistent

trend with curing was observed between the NaOH-2 h and NaOH-24 h extractable OC, i.e. the NaOH-2 h extractable OC was not necessarily more degradable than the NaOH-24 h extractable OC (Table 5-2).

As discussed earlier, total extractable OC in each extraction decreased with curing (Table 5-2). The reduction was more pronounced in samples with high extractable OC such as RC samples. Decrease of water-extractable OC with composting has been reported (Bernal et al., 1998b; Chefetz et al., 1998b; Hue and Liu, 1995). Hue and Liu (1995) suggested to use 10 g water-extractable OC per kg dry matter as the cut-off value for stable compost. Using this value to screen the compost samples in the current study, similar conclusion to that based on CO_2 evolution rate was obtained, i.e. all compost samples except for RC were stable (Table 5-2).

Composting is a humification process, thus, concentration of humic substance is expected to increase with composting. Inconsistent changes in NaOH-extractable HA have been reported (Bernal et al., 1998b; Calace et al., 1999; Pascual, et al., 1997; Sanchez-Monedero et al., 1999). On the other hand, significant and consistent reduction in FA concentrations was observed almost in all studies. Our results were consistent with those reported in the literature. Though, the ratios of HA/FA generally increased with curing primarily due to the consistent and substantial reduction of FA fraction, such changes were inconsistent, which agreed well with data published by Bernal et al. (1998b). This was most likely because our samples were in a relative short curing stage (< 30 day). Thus, changes in the ratio of HA/FA during this period were probably insignificant. The ratios of HA/FA also varied greatly with composting source materials, and seemed to be less correlated with other parameters measuring compost stability.

Similar to changes in extractable OC, the ratios of HA/FA increased in the order of water-2h (0.11- 0.99), NaOH-2h (0.41-3.48), and NaOH-24 h (1.03-4.01) as the extraction intensity increased. These three fractions consisted of OC of different degree of polymerization and aromaticity, which can be measured based on photometric properties using MSA. The greater the MSA, the higher the degrees of polymerization and aromaticity. Thus, as expected, the MSA of HA is greater than FA, and MSA of the NaOH-extractable OC (HA and FA) is greater than that of the water-extractable OC (Table 5-3). With a few exception, MSA of HA and FA increased with curing (Table 5-3), indicating humification was occurring during curing of those composts.

There were nine fractions of OC including three extractions with each contained HA, FA and sum of HA+FA fractions. Carbon concentrations in different fractions were positively correlated to each other, with correlation coefficients (r) ranging from 0.56 to 0.98, with a few exceptions (data not shown). The correlation between the NaOH-extractable HA and NaOH-extractable FA (0.56 and 0.76 for NaOH-2h and 24h), respectively, was greater than those between the water-extractable HA and water-extractable FA (R^2=0.56). The fairly high concentration of FA in the NaOH extractable fraction seems to support the hypothesis that FA might not be a truly independent part of humic substance, rather it is a partial product of hydrolysis of HA during the NaOH extraction (Orlov, 1999). Longer reaction time and more vigorous shaking during the extraction procedure may have resulted in more FA compared to the actual amount of FA present.

Table 5-3. Mass specific absorbance of water and NaOH extractable FA and HA (L mg $^{-1}$m^{-1}).

Compost	Curing	Water		NaOH-2 h		NaOH-24 h	
	(d)	FA	HA	FA	HA	FA	HA
RC-1	0	0.06 ± 0.00	0.33 ± 0.07	0.09 ± 0.00	0.90 ± 0.05	0.09 ± 0.00	0.85 ± 0.01
RC-1	7	0.09 ± 0.01	0.44 ± 0.05	0.11 ± 0.00	1.18 ± 0.02	0.10 ± 0.01	1.13 ± 0.02
RC-1	30	0.10 ± 0.00	0.68 ± 0.07	0.12 ± 0.01	1.39 ± 0.08	0.11 ± 0.00	1.24 ± 0.05
RC2	0	0.08 ± 0.00	1.06 ± 0.56	0.10 ± 0.00	0.93 ± 0.00	0.09 ± 0.00	0.96 ± 0.00
RC2	9	0.07 ± 0.00	0.64 ± 0.00	0.11 ± 0.00	0.88 ± 0.00	0.10 ± 0.01	1.00 ± 0.20
RC2	21	0.10 ± 0.00	0.49 ± 0.02	0.14 ± 0.00	1.41 ± 0.02	0.13 ± 0.01	1.46 ± 0.11
RC2	25	0.13 ± 0.00	0.46 ± 0.04	0.16 ± 0.01	1.30 ± 0.04	0.11 ± 0.01	1.34 ± 0.04
WPB	0	0.09 ± 0.00	0.23 ± 0.02	0.12 ± 0.02	0.93 ± 0.05	0.07 ± 0.01	0.89 ± 0.01
WPB	7	0.10 ± 0.00	0.31 ± 0.03	0.12 ± 0.00	1.30 ± 0.39	0.09 ± 0.01	1.00 ± 0.06
WPB	30	0.15 ± 0.00	0.94 ± 0.05	0.18 ± 0.02	1.58 ± 0.10	0.13 ± 0.01	1.38 ± 0.02
SA	0	0.10 ± 0.00	0.71 ± 0.01	0.10 ± 0.00	0.62 ± 0.09	0.09 ± 0.00	0.61 ± 0.07
SA	14	0.10 ± 0.00	0.80 ± 0.02	0.11 ± 0.00	1.04 ± 0.22	0.08 ± 0.00	0.59 ± 0.12
SA	30	0.12 ± 0.00	1.14 ± 0.07	0.12 ± 0.01	0.74 ± 0.03	0.09 ± 0.00	0.66 ± 0.04
MW	30	0.07 ± 0.00	0.40 ± 0.00	0.11 ± 0.01	0.38 ± 0.03	0.08 ± 0.00	0.45 ± 0.01
MW	90	0.10 ± 0.01	1.26 ± 0.26	0.16 ± 0.01	0.83 ± 0.08	0.10 ± 0.02	0.69 ± 0.04
PR	0	0.05 ± 0.00	0.84 ± 0.06	0.14 ± 0.00	0.72 ± 0.03	0.13 ± 0.00	0.72 ± 0.01
PR	7	0.06 ± 0.00	0.45 ± 0.00	0.15 ± 0.00	0.90 ± 0.08	0.13 ± 0.01	0.88 ± 0.09
PR	14	0.05 ± 0.00	0.41 ± 0.04	0.16 ± 0.01	1.36 ± 0.07	0.17 ± 0.01	1.01 ± 0.07

Relation of Extractable Organic Carbon to CO_2 Evolution Rate

Concentration of OC in each of the nine fractions was highly and linearly correlated to CO_2 evolution rate with R^2 = 0.35-0.79 ($p<0.01$), except for the water-2h HA (R^2=0.27) (data not shown). The NaOH-2 h FA had the highest correlation coefficient (R^2=0.95, $p<0.001$) followed by the water-2h FA fraction (R^2=0.89 $p<0.001$) (Fig. 5-1). The combination of the water-2h FA and NaOH-2 h FA was also highly correlated with CO_2 evolution rate, with R^2=0.93 ($p<0.001$). Thus, concentrations of FA can be used to indicate compost stability. In addition, the FA fraction can be easily measured by a simple photometric method, since the MSA of FA fraction is within a relative narrow range of 0.05-0.18 L mg $^{-1}$m^{-1} (Table 5-3). In fact, FA fraction was highly correlated to its absorption with R^2=0.95 ($p<0.001$) for the water-2h FA and R^2=0.87 (0.001) for NaOH-2h FA fraction (Fig. 5-2). Concentrations of OC in these compost extracts covered a wide range up to 4 g L^{-1} C, which makes it possible to determine OC concentration in a simple fashion.

To further understand the contribution of each fraction of OC to CO_2 evolution rate, a stepwise multiple regression procedure was used. The regression equation ($p<0.01$) is as follows:

$$CO_2 = -0.252 + 0\,013FA_{water\text{-}2h} - 0.037HA_{water\text{-}2h} + 0.025FA_{NaOH\text{-}2h} + 0.013HA_{NaOH\text{-}2h} \quad \text{Eq. 5-1}$$

Based on Eq.5-1, water-2h and NaOH-2 h extractable OC accounted for 95% ($p<0.01$) of the variability, indicating the NaOH-24 h extractable OC was insignificant in contributing to CO_2 evolution. It may also indicate that the NaOH-24 h extractable OC was either stable humic substance or was a lignin-type material that was resistant to decomposition, although the percentage of total extractable OC was the highest for this fraction.

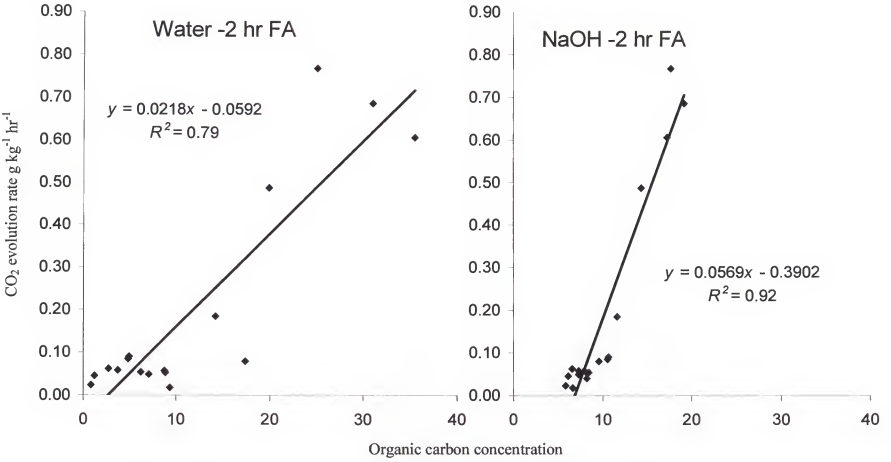

Figure 5-1. Relationship between organic carbon concentration of FA fraction and compost CO_2 evolution.

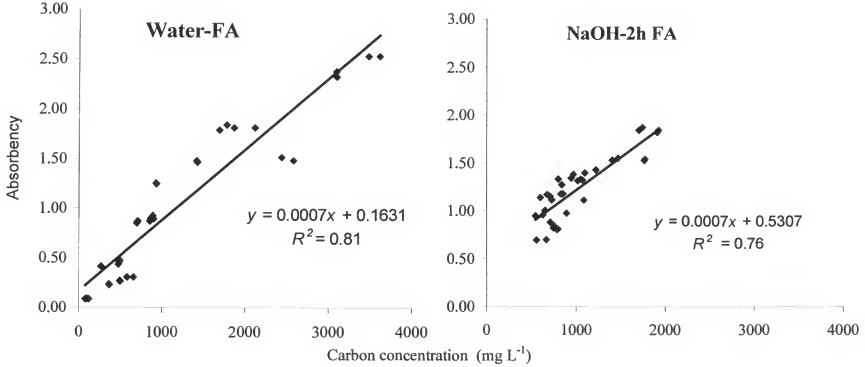

Figure 5-2. Relation of carbon concentration of water-2h FA and NaOH-2h FA with their absorbence at 420 nm.

Water-2h extractable HA had a negative effect on CO_2 evolution (Eq. 5-1), while both NaOH fractions contributed positively to CO_2 evolution. Based on fractionation and NMR data, Chefetz et al. (1998b) concluded that one fraction of water extractable HA was related to real HA, which represents an intermediate state in the humification process. The negative correlation between CO_2 evolution rate and OC content of this fraction leads to the speculation that the water-2h HA fraction was not a carbon source for microbial growth. Instead, it may ultimately polymerize and precipitate into water-insoluble HA as the compost further matures.

Ratio of HA/FA was not significantly related to CO_2 evolution rate when all samples were combined, although it was significantly ($p<0.01$) correlated to CO_2 evolution rate for RC samples (data not shown). Similar observation was made by Jimenez and Garcia (1992) and Bernal et al. (1998b) and they attributed this inconsistency to dependency of this parameter on the differences in compost source materials.

Summary

Extractable OC decreased with curing, which was influenced by the composition of compost source materials. Except for water-2h HA, OC contents in the FA and HA fractions of each extraction were correlated to each other and CO_2 evolution rate, but multiple regression showed that the NaOH-24 hr fractions was insignificant in contributing to CO_2 evolution. Water-2h FA and/or NaOH-2 hr FA are recommended as indicators for compost stability due to their high correlation with compost stability estimated by CO_2 evolution. Concentrations of FA fraction can be estimated by a simple photometric method for a large range of C concentration

CHAPTER 6
EFFECTS OF COMPOST STABILITY AND APPLICATION RATE ON CARBON AND NITROGEN MINERALIZATION WHEN INCUBATED WITH SOIL

Introduction

Evaluation of composts for land application purpose has focused on using compost stability as an index to determine compost quality. Recently mineralization rate of compost has become a bigger concern (Hadas and Portnoy, 1997). Understanding C and N mineralization, especially the ability and timing of compost to provide N and the fate of N in soil, is necessary to optimize fertilizer response and to minimize nitrate N leaching (Smith et. al., 1998).

Effects of compost stability on the dynamics of C and N mineralization have been studied, using composts of different source materials or composts at different composting stages (Hadas and Portnoy, 1994; Keeling et al., 1995; Bernal et al., 1998 a, c). Unstable compost generally has a higher decomposition rate and thus releases more CO_2 (Hadas and Portnoy, 1994). The effects of compost stability on N dynamics depend on the compost C/N ratio and total nitrogen content. Unstable compost with high available C and a wide C/N ratio may lead to a net reduction in the mineral N content in soils due to immobilization (Epstein et al., 1978) causing plant N deficiency (Bernal et al. 1998a). Sommers (1983) estimated that the potential for N immobilization was greatest when the C/N ratio of the biosolids exceeded 20:1. On the other hand, Kaboneka et al. (1997) suspected the critical C/N ratio might be lower than suggested. Stable composts usually have a low C/N ratio, which makes net nitrification likely to occur. Mature compost with

nitrate accumulation can be added to a soil when the crop is growing (Bernal et al. 1998a), but has a greater potential of nitrate leaching risk. By studying 12 biosolids with different treatment modes, Smith et al. (1998) concluded that digested biosolids have a greater potential of nitrate accumulation than undigested biosolids because of the higher NH_4-N concentration. He also pointed out that the dominant factors controlling the rate and extent of NO_3 formation in amended soil were biosolids type (stability of organic matter), soil temperature and time of soil incorporation.

The effects of compost or biosolids application rate on C and N dynamics have been studied less extensively. This is probably because the effects of application rate is usually masked by factors such as crop N need and pollutant or soluble salts content in the compost. The effects of application rate on C and N behavior seems to be of less interest. Yet, as Hadas and Portnoy (1997) pointed out, the recovery of CO_2 evolution depends on application rate and to a less extent on the soils, which means the mineralization of C and N are closely related to application rate. Consequently, application rate might be an important parameter in terms of controlling of C and N behavior after organic waste addition to a soil.

Materials and Methods

Soil and Compost Samples

Biosolids compost samples used in this experiment were RC-1 and RC-3 samples as described in Chapter 2. The two samples used in this study were at 1day and 30 day of curing. The soil used was an Ultisol (Arredondo, grossarenic paleudult) of loamy texture from Florida. Both soil and compost samples were air dried. The selected physical and chemical properties of the soil and composts are listed in Table 6-1. The soil pH and EC

Table 6-1. Selected physical and chemical properties of soil and compost samples.

	pH	EC ($ms\ cm^2\ g^{-1}$)	Organic carbon ($g\ kg^{-1}$)	N ($g\ kg^{-1}$)	C/N
Soil	5.8	0.18	12	1	12
RC-1	5.8	49.1	398	28	14
RC-3	8.4	42.0	392	26	15

were measured in 1:2 solid to water ratio extract. The extraction ratio of compost was 1:10.

Experimental Design and Parameters Measured

Compost samples were mixed with soil at compost to soil dry mass ratio of 1:44, 1:8.8 and 1:4.4, corresponding to application rates of 50, 250 and 500 Mg ha^{-1}, assuming the top 15cm of surface soil weights 2.2 $x10^6$ g. Experimental controls included both a soil control and compost control. Each treatment was repeated 15 times. WHC of soil and compost mixture was estimated by the crude method using a funnel. Samples were placed in 120 ml Corning brand snap-seal plastic containers with the lid open to allow air exchange. The moisture content during incubation was adjusted to about 60% of WHC of each treatment. The samples were randomized and kept at room temperature in dark. Three replicates of each treatment were taken at 5, 13, 25, 50, 85 day. Water was added regularly based on the weight loss to maintain the moisture content. During the incubation process, CO_2 evolution was measured every few days depending on the microbial activity. After each sampling, the soil and compost samples were extracted with water at solid to water ratio of 1:10 for compost and 1:2 for soil for 2 h at a horizontal shaker. For the soil+compost mixture the extraction was conducted in such a way that the compost in the mixture were extracted at 10:1 water to compost ratio. This was achieved by adding the amount of water to the water holding capacity of soil plus 10

times of compost mass in the mixture. It is acknowledged that this method is only a rough way to keep the extraction ratio similar for compost among treatments. The rational of doing so is that water extractable substances from soil is insignificant compared to that from compost. When comparing C and N mineralization of compost, it is more reasonable keeping consistent extraction ratio based on compost rather than the total mass, otherwise, the low application rate treatment would have much higher extraction rate for compost than that of high application rate treatment.

WSOC was measured as in Chapter 2. Water soluble NH_4^+ and NO_3^- were determined after the water extract were adjusted to pH<2 and part of water soluble organic matter precipitated out and was removed by centrifuging at 9,630 for 10 min as the high concentration of water soluble organic matter interfere the NH_4^+ and NO_3^- analysis. NH_4^+ was measure by EPA method 350.1 and NO_3^- and NO_2^- were measured by EPA method 353.2. Since the incubation system was aerated, NO_3^- was considered the major component.

Statistical Analyses

The SAS program was used to detect statistically significant differences ($P<0.05$) between treatments (SAS, 1987). A Proc CORR procedure was used to compute the linear relation coefficient between variables.

Results and Discussion

pH and EC

The pH of soil alone treatment didn't exhibit clear change during incubation, that of amended soil decreased and compost alone increased, corresponding to the decrease of

Table 6-2. pH of each treatment at each sampling in 1:10 compost to water ratio extract.

	5d	13d	25d	50d	85d
Soil	6.68 ± 0.15†	6.83 ± 0.23	6.36 ± 0.07	6.79 ± 0.04	6.26 ± 0.07
RC-1-50t/ha	7.06 ± 0.02	7.12 ± 0.11	6.13 ± 0.18	6.95 ± 0.05	5.68 ± 0.19
RC-1-250t/ha	7.56 ± 0.10	7.67 ± 0.04	7.34 ± 0.07	6.94 ± 0.00	5.93 ± 0.13
RC-1-500t/ha	7.81 ± 0.04	7.85 ± 0.02	7.63 ± 0.02	7.02 ± 0.02	6.49 ± 0.21
RC-1	6.97 ± 0.01	7.16 ± 0.05	7.62 ± 0.06	7.21 ± 0.03	8.28 ± 0.03
RC-3-50t/ha	6.84 ± 0.15	7.00 ± 0.05	6.09 ± 0.30	6.69 ± 0.18	5.84 ± 0.10
RC-3-250t/ha	7.52 ± 0.01	7.48 ± 0.06	6.38 ± 0.06	6.40 ± 0.23	5.25 ± 0.07
RC-3-500t/ha	7.85 ± 0.05	7.44 ± 0.03	6.81 ± 0.10	6.53 ± 0.02	5.19 ± 0.05
RC-3	8.33 ± 0.04	7.85 ± 0.04	7.49 ± 0.07	7.19 ± 0.02	7.73 ± 0.01

† One standard deviation of three replicates.

Table 6-3. EC (normalized to dry mass unit of mixture of compost and soil) of each treatment at each sampling (ms cm^2 g^{-1}).

	5d	13d	25d	50d	85d
soil	0.2 ± 0.01†	0.3 ± 0.00	0.6 ± 0.04	0.7 ± 0.10	0.7 ± 0.04
RC-1-50t/ha	0.8 ± 0.06	0.8 ± 0.18	2.1 ± 0.00	1.5 ± 0.06	1.7 ± 0.03
RC-1-250t/ha	4.2 ± 0.24	3.9 ± 0.14	4.3 ± 0.14	6.1 ± 0.14	7.1 ± 0.37
RC-1-500t/ha	8.2 ± 0.06	7.6 ± 0.06	7.3 ± 0.15	7.6 ± 0.35	9.6 ± 0.71
RC-1	47.6 ± 1.64	46.7 ± 1.42	44.6 ± 2.55	39.7 ± 2.61	43.3 ± 1.53
RC-3-50t/ha	0.7 ± 0.00	0.7 ± 0.07	1.1 ± 0.00	1.4 ± 0.02	1.4 ± 0.03
RC-3-250t/ha	3.8 ± 0.15	3.4 ± 0.07	5.4 ± 0.10	6.8 ± 0.02	7.7 ± 0.10
RC-3-500t/ha	7.1 ± 0.12	6.9 ± 0.09	8.2 ± 0.22	10.1 ± 0.00	10.9 ± 0.11
RC-3	41.3 ± 1.90	41.2 ± 0.95	39.6 ± 0.35	38.7 ± 0.35	38.6 ± 1.37

† One standard deviation of three replicate

NH_4^+ and accumulation of NO_3^- (Table 6-2, 6 and 7). I speculated that in this system the main factors that influence pH include NH_4^+ concentration, acidity produced by nitrification process, and organic acid concentration (indicated by WSOC) present in compost. It is well known that high concentrations of NH_4^+ will increase soil pH and nitrification process will decrease soil pH. Less known is the possible effect of organic acids on soil pH. It is reported that in unstable compost, high concentration of organic acids lead to low pH even at high NH_4^+ concentration (Avnimelech et al., 1996), as can be seen from RC-1 vs. RC-3 sample at the early stage of incubation. During incubation, the organic acids degraded and the pH difference disappeared between the two.

EC decreased in the treatment of compost alone, probably due to the decrease of NH_4^+ and WSOC, but increased in other treatments as the nitrification process occurred. EC values were highly correlated ($p<0.01$, $R^2=0.96$) with inorganic nitrogen concentration ($NH_4^+ + NO_3^-$) for all data points combined. It has been reported that 0.8 fold increase of NO_3^- lead to 1.5 unit increase of EC in a California soil (Stamatiadis et al., 1999). Doran et al. (1996) also demonstrated the predictive capability of soil EC to estimate soil NO_3^- concentration. In this system, due to the high application rate of compost, although the contribution of NO_3^- to EC was significant, factors such as NH_4^+ and other soluble salts were also important. As a result, EC was highly correlated with total inorganic N instead of NO_3^- alone.

Carbon Mineralization (CO_2 evolution)

The estimates of compost-C mineralized over the incubation was made by subtracting the amount of released CO_2 from soil control from that of each treatment (Fig 6-1). This assumes compost was the sole source of increase in total CO_2 emitted with compost

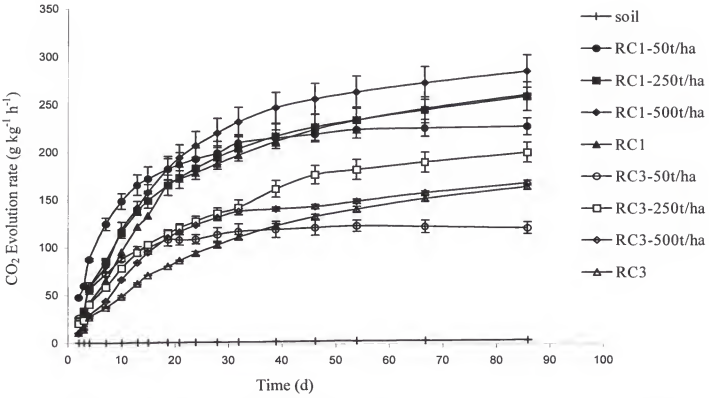

Figure 6-1. Cumulated CO_2 evolution from per mass unit of soil or compost incubated with soil at room temperature.

application. The cumulated CO_2 evolution curve can be grouped according to compost, which indicates the effect of compost stability on C mineralization. CO_2 evolution from the less stable RC-1 sample, with or without soil, is higher than that from RC-3 sample. This result is expected as the younger compost contains more degradable organic carbon (Bernal et al, 1998c; Hadas and Portnoy, 1994). In the early stage of incubation, the low application rate treatment of 50 t ha^{-1} had the highest and the compost alone had the lowest CO_2 evolution for both RC-1 and RC-3 samples, which indicates that the organic C in compost degraded faster at low application rate. Consequently, the low rate treatment reached the plateau of CO_2 evolution earlier than other treatments. Hadas and Portnoy (1997) reported that 13-15% and 8% of compost C recovered as CO_2 for the low and high application rates after 33 wk of incubation with soil, which partially confirmed our results. No clear explanation for this trend can be found from the literature. I suspected that the smaller recovery of C as CO_2 at the high compost level could be due to the higher microbial use efficiency (fraction of available C used for microbial growth) at high available C concentration (Hadas and Portnoy, 1997), thus less C released as CO_2.

Water Soluble Organic Carbon (WSOC)

In order to exam the effect of compost on the WSOC of soil, WSOC was expressed on the basis of per unit of total dry mass. WSOC decreased with incubation time for all the treatments, including compost and soil control. The possible reason that WSOC of soil alone decreased over time is the disturbance of soil increased the WSOC at the beginning of incubation, with time soil WSOC was utilized by the microbes. Except for the late stage of 50 t ha^{-1} application rate, WSOC of compost alone and compost amended soil were higher than that of the soil control, demonstrating the impact of compost application on WSOC at high application rate. WSOC were consistently higher

Table 6-4. Total water extractable organic carbon concentration (mg kg^{-1} dry mass of compost and/or soil) during incubation.

	5d	13d	25d	50d	85d
Soil	83 ± 5†	36 ± 2	25 ± 6	14 ± 2	19 ± 1
RC-1-50t/ha	388 ± 12	204 ± 3	82 ± 6	47 ± 118	45 ± 1
RC-1-250t/ha	2293 ± 36	1969 ± 121	1550 ± 51	889 ± 96	579 ± 7
RC-1-500t/ha	4685 ± 477	4579 ± 69	4306 ± 161	3532 ± 457	2313 ± 199
RC-1	36421 ± 407	33858 ± 627	28978 ± 1140	24996 ± 1510	27280 ± 799
RC-3-50t/ha	232 ± 24	97 ± 2	31 ± 3	33 ± 0	28 ± 1
RC-3-250t/ha	1770 ± 98	1421 ± 64	778 ± 28	568 ± 62	338 ± 13
RC-3-500t/ha	3762 ± 110	3395 ± 16	2430 ± 51	1414 ± 23	959 ± 24
RC-3	23269 ± 765	19767 ± 983	16820 ± 587	16886 ± 457	16048 ± 576

† One standard deviation of three replicate

in treatments with RC-1 sample than of RC-3 sample, a trend in agreement with the CO_2 evolution. For the first sampling, WSOC of compost amended soils are roughly proportional to the compost application rate. As incubation progressed, the differences among different application rates widened, showing the low rate treatment decreased faster in WSOC, especially the 50 t ha^{-1} treatment. By 25 d and 13d, the WSOC of RC-1 and RC-3 were close to the level of soil (82 mg kg^{-1}) at first sampling, which suggests the impact of compost on WSOC were negligible by that time. This observation corresponds well with the CO_2 evolution data in that it was approximately the same time when the accumulated CO_2 evolution curves leveled off (Fig. 6-1). The relation between WSOC and CO_2 production was not consistent. Burford and Bremner (1975) and Davidson et al. (1987) found a strong correlation between WSOC concentrations and C mineralization in a variety of soil types. Cook and Allan (1992) reported that there was no obvious relationship between WSOC and C mineralization rate over a long incubation period, but they found a positive association between WSOC and C mineralization rate at early incubation times. Under field condition, Gregorich et al. (1998) found that WSOC was not a significant predictor of CO_2 flux because factor other than WSOC limit soil respiration at higher manure application rates.

Nitrogen Mineralization and Nitrification

In general, inorganic nitrogen (NH_4^+ and NO_3^-) concentration decreased with incubation, indicating the loss of inorganic N, either through NH_4^+ volatilization or N immobilization in the early stage of incubation and denitrification of NO_3^- at later stage. For all treatments, NH_4^+ concentration decreased, while NO_3^- concentration increased and then decreased for some treatments over time. Nitrification occurred earlier in treatments with RC-3 than in RC-1 sample. The time sequence of NO_3^- appearance among different

Table 6-5. Inorganic nitrogen concentration (NH_4^+-N +NO_3^-, mg kg^{-1} dry mass of compost and/or soil) during incubation.

	5d	13d	25d	50d	85d
Soil	11 ± 3†	28 ± 1	72 ± 4	89 ± 16	74 ± 6
RC-1-50t/ha	50 ± 4	64 ± 10	260 ± 9	170 ± 38	164 ± 13
RC-1-250t/ha	294 ± 67	277 ± 37	399 ± 18	589 ± 135	757 ± 199
RC-1-500t/ha	640 ± 60	586 ± 75	566 ± 26	641 ± 33	983 ± 133
RC-1	4544 ± 674	4971 ± 120	3944 ± 956	3080 ± 183	3927 ± 73
RC-3-50t/ha	35 ± 6	64 ± 2	229 ± 132	150 ± 15	140 ± 31
RC-3-250t/ha	275 ± 29	236 ± 23	451 ± 80	766 ± 29	1156 ± 519
RC-3-500t/ha	534 ± 40	508 ± 22	642 ± 203	1007 ± 31	798 ± 271
RC-3	3729 ± 571	3241 ± 113	2762 ± 70	2592 ± 59	2611± 446

† One standard deviation of three replicate

Table 6-6. NH_4^+-N concentration (mg kg^{-1} mg kg^{-1} dry mass of compost and/or soil) in each treatment during incubation.

	5d	13d	25d	50d	85d
Soil	5 ± 2†	3 ± 1	2 ± 2	1 ± 0	1 ± 0
RC-1-50t/ha	50 ± 4	46 ± 8	7 ±1	1 ± 0	0 ± 0
RC-1-250t/ha	294 ± 67	277± 37	312 ± 12	193 ± 31	185 ± 71
RC-1-500t/ha	640 ± 60	586 ± 75	565 ± 27	490 ± 24	462 ± 18
RC-1	4544 ± 674	4971 ± 120	3944 ± 956	3080 ± 183	3873 ± 64
RC-3-50t/ha	34 ± 7	33 ± 1	4 ± 2	1 ± 0	0 ± 0
RC-3-250t/ha	275 ± 29	236 ± 23	167 ± 21	73 ± 26	5 ± 0
RC-3-500t/ha	534 ± 40	508 ± 22	386 ± 99	148 ± 16	7 ± 1
RC-3	3705 ± 544	3241 ± 113	2762 ± 70	2587 ± 58	2472 ± 434

† One standard deviation of three replicate

Table 6-7. NO_3^--N concentration (mg kg^{-1} dry mass) in each treatment with incubation.

	5d	13d	25d	50d	85d
Soil	7 ± 0†	26 ± 1	70 ± 4	88 ± 16	73 ± 6
RC-1-50t/ha	0 ± 0	18 ± 2	253 ±	169 ± 38	164 ± 13
RC-1-250t/ha	0 ± 0	0 ± 0	87 ± 9	397 ± 107	572 ± 131
RC-1-500t/ha	0 ± 0	0 ± 0	0 ± 1	151 ± 29	522 ± 150
RC-1	0 ± 0	0 ± 0	0 ± 0	0 ± 0	53 ± 17
RC-3-50t/ha	1 ± 1	30 ± 1	226 ± 130	150 ± 15	140 ± 32
RC-3-250t/ha	0 ± 0	0 ± 0	283 ± 59	693 ± 47	1151 ± 518
RC-3-500t/ha	0 ± 0	0 ± 0	256 ± 105	859 ± 47	791 ± 270
RC-3	24 ± 29	0 ± 0	0 ± 0	5 ± 2	139 ± 52

† One standard deviation of three replicate for each treatment.

compost treatment was soil alone >50 t/ha >250 t/ha >500 t/ha > compost alone. It took the high application rate treatment longer for nitrification to occur. It is reported that high NH_4^+ concentration at 400 mg kg^{-1} (McIntosh and Frederick, 1958) to 800 mg kg^{-1} (Broadbent et al., 1957) could inhibit the nitrification process. The result of this study does not support this finding because nitrification occurred in the compost alone treatment at the end of the incubation with NH_4^+ concentration >2000 mg kg^{-1}. On the other hand, nitrification didn't happen at the early stage of incubation even when NH_4^+ concentration was as low as <50 mg kg^{-1} for the 50t/ha treatment of RC-1 sample. I suspect that the inhibition of nitrification at early stage of incubation is a result of competition between the heterotrophic and autotrophic bacteria as the growth rate of nitrifying bacteria is about one fourth that of carbon oxidising heterothophic bacteria (Aesoy et al., 1998; Cheng and Chen, 1994). When available carbon is present, nitrification is more likely to be suppressed. In a waste water treatment system, Figueroa and Silverstein (1992) found that particulate organic matter restrained the nitrification rate at the same extent as dissolved organic matter. Andersson et al. (1994) found a more than 40% increase in the nitrification activity in a trickling filter when the concentration of suspended solids was reduced to 15 g suspended solids L^{-1}. This conclusion is also partially confirmed by the fact that nitrification occurred earlier with RC-3 under the same application rate, because RC-3 compost had less available organic carbon compared to RC-1 sample, even though the two samples have very similar C/N ratios. However, nitrification was not linearly related to WSOC concentration. Nitrification still occurred after certain period of time at high WSOC level (Table 6-4 & 6-7). This suggests that either WSOC might not be a good indicator of easily available organic carbon or other

chemicals in the system might be toxic to the nitrification process. On the other hand, the result also showed that application rate was important in regulating nitrification, along with other factors such as compost stability, C and N availability.

Summary

Incubation of compost with soil enhanced compost degradation compared to compost alone without soil, and the enhancement was more effective at low compost application rate. Less stable compost released more carbon as CO_2 and maintained a higher NH_4^+ concentration in the system. Nitrification occurred earlier with more stable compost. Low application of compost is favorable for occurrence of nitrification, probably due to the lower concentration of WSOC, yet their relation remained unclear. Based on Compost C and N dynamics are the functions of compost stability. Application rate also had an effect on the result of N dynamics, as it influences the occurrence of nitrification.

CHAPTER 7
CONCLUSIONS

Compost products vary in quality due to the heterogeneous nature of compost source materials and composting process. Among the numerous parameters measuring compost quality are the compost stability and maturity, which are mainly used to indicate the degree of organic matter degradation and readiness of compost to be used. Crop damages due to insufficient composting have been reported. Compost stability and maturity have become an issue and topic of scientific debate in the industry and research community. Without a clear understanding of the two concepts, it is difficult to develop a universal parameter on which industry standards can be based. The current study was mainly focused on biosolids compost. However, the compost samples used in this study were taken from full scale composting facilities and covered a wide range of other materials to co-compost with biosolids. In addition, the methods used in the study were not dependent on, or confined to biosolids compost. Thus, it is reasonable to expect the findings of the study may be applicable to other composts.

The first objective of this study was to compare several methods assessing compost stability and maturity, aiming to understand the difference and relation between the two concepts, and attempting to identify a simple and reliable method for determining stability and maturity of biosolids compost. The results indicated compost stability indicated by CO_2 evolution and maturity indicated by seed germination are indeed two different characteristics of compost quality. Stability and maturity may be correlated, e.g. more stable compost tends to be more mature. However, some stable composts may not

be mature because they need more time for microbes to break down the phytotoxic substances due to the nature of compost source material or compost process. On the other hand, 'mature' compost may have a relatively high microbial activity without showing phytotoxicity. Both parameters, stability and maturity, are needed to assure a quality compost product. Water soluble organic carbon (WSOC) concentration was correlated to CO_2 evolution and germination rate.

The second objective was to study the effects of sample storage on three test methods of evaluating compost stability and maturity due to the research need and the fact that composting facilities may not be able to perform all the analyses on site. Compost samples were stored air-dry and frozen for one year. Depending on the stability and maturity level of the compost and on the compost feedstock source, air-drying and freezing samples had varied effects on the three measured parameters: CO_2 evolution rate, seed germination rate, and WSOC concentration. Neither of the two storage methods was satisfactory in terms of maintaining the original properties of the fresh compost. Storage tended to delay the CO_2 evolution of the active sample, leading to lower accumulated CO_2 evolution during the test time. Peak CO_2 evolution rate was thus a more suitable parameter for compost samples with high microbial activity. It made no significant difference for more stable composts as they had lower CO_2 evolution and did not exhibit notable peaks. It is possible to preserve compost samples for phytotoxicity analysis, nevertheless fresh samples without storage is recommended for phytotoxicity test until the storage effect is fully understood. Compared to fresh samples, air-dry storage reduced and frozen storage increased the WSOC concentrations of compost samples. The storage effect was not significant on compost with low initial WSOC.

Despite all these variations, WSOC has a significant and consistent correlation with both CO_2 evolution and seed germination.

The third objective was closely related to what was found with the first and second objectives, i.e. assessing the feasibility of using WSOC to indicate compost stability. Based on six batches of compost samples from five different composting facilities, OC contents in the FA and HA fractions of each extraction, except for water-2h HA, were correlated to each other and CO_2 evolution rate. However, multiple regression showed NaOH-24 hr fractions were insignificant in contributing to CO_2 evolution. Water-2h FA and/or NaOH-2 hr were highly correlated to CO_2 evolution, indicating they can be used to predict compost stability. Concentrations of FA carbon can be estimated by a simple photometric method over a large range of C concentration.

The fourth objective of this study was to investigate the effect of compost stability and application rate on C and N mineralization. Incubation of compost with soil enhanced compost degradation compared to compost alone without soil, and the enhancement was more obvious at low compost application rate. Less stable compost released more carbon as CO_2 and maintained a higher ammonium concentration in the system. Nitrification occurred earlier with more stable compost. Low application of compost was favorable for nitrification, probably due to the lower concentration of WSOC, yet their relation remained unclear. The results indicate that compost C and N dynamics are a function of compost stability. Application rate also had a great effect on the result of N dynamics, as it influenced the nitrification process. This information may be very helpful in managing land application of compost in an economical and environmental sound manner.

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BIOGRAPHICAL SKETCH

Lingzheng Wu was born in Zhejiang Province, China. She graduated from Beijing Agricultural University with a B. S. in 1991. Then she went on to to get her M. S. in 1991 from the same university. Her major area of study involved predicting soil Cd contamination with plant seedlings. In the following two years, she worked for the Research Center of Ecological and Environmental Science on the heavy metal adsorption and desorption from a contaminated river sediment resulting from copper mining. In 1996, she started work on a Doctor of Philosophy degree under the direction of Dr. Lena Ma in the Soil and Water Science Department at University of Florida, investigating stability and maturity evaluation of biosolids compost.

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

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